
**Hoy's Marine Site
Newport, Oregon
Sampling and Quality Assurance Plan
TDD: 98-07-0011**

Contract: 68-W6-0008
April 1999

Region 10
START

Superfund Technical Assessment and Response Team

Submitted To: Dave Bennett, Task Monitor
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SAMPLING AND QUALITY ASSURANCE PLAN FOR:

**Hoy's Marine Site
Newport, Oregon
TDD: 98-07-0011**

Prepared By:

Ecology and Environment, Inc.

Contract No: 68-W6-0008

Date: April 1999

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SQAP DISTRIBUTION LIST

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LIST OF ACRONYMS

Al ₂ O ₃	Aluminium Oxide
bgs	Below the Ground Surface
CLP	Contract Laboratory Program
CLPAS	Contract Laboratory Program Analytical Service
DBT	Dibutyltin
DQO	Data Quality Objective
E&E	Ecology and Environment, Inc.
EPA	Environmental Protection Agency
ERL	Effects Range-Low
Fe ₂ O ₃	Ferric Oxide
FOWP	Field Operations Work Plan
GEM	GEM Consulting, Inc.
GPS	Global Positioning System
HAZWOPER	Hazardous Waste Operation
MBT	Monobutyltin
MFA	Maul Foster & Alongi, Inc.
MgO	Managanese Oxide
NFIP	National Flood Insurance Program
NOAA	National Oceanic and Atmospheric Administration
ODEQ	Oregon Department of Environmental Quality
ODFW	Oregon Department of Fish and Wildlife
ONHP	Oregon Natural Heritage Program
PAH	Polycyclic Cromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PM	Project Manager
PPE	Probable Point of Entry
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QMP	Quality Management Plan

LIST OF ACRONYMS (CONTINUED)

<u>Acronym</u>	<u>Definition</u>
RSCC	Regional Sample Control Coordinator
SI	Site Inspection
SiO ₂	Silicon Oxide
SOP	Standard Operating Procedure
SQAP	Sampling and Quality Assurance Plan
SRH	SRH Associates, Inc.
START	Superfund Technical Assessment and Response Team
SVOC	Semi-Volatile Organic Compound
TAL	Target Analyte List
TBT	Tributyltin
TCLP	Toxicity Characteristic Leaching Procedure
TDD	Technical Direction Document
TM	Task Monitor
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbon
USDA	United States Department of Agriculture
USDI	United States Department of the Interior
USGS	United States Geological Survey
UST	Underground Storage Tank
VOC	Volatile Organic Compound
WRCC	Western Regional Climate Center

**SAMPLING AND QUALITY ASSURANCE PLAN FOR:
HOY'S MARINE SITE
NEWPORT, OREGON**

TDD: 98-07-0011

1. PROJECT MANAGEMENT

1.1 PROJECT/TASK ORGANIZATION

This section outlines the individuals directly involved with the Hoy's Marine site project and their specific responsibilities. Lines of communication are shown in the Project Organization Chart (Figure 1-1).

1.1.1 Environmental Protection Agency (EPA) Region 10 Task Monitor (TM)

Overall coordinator of the project and decision maker. Reviews and approves the site-specific Sampling and Quality Assurance Plan (SQAP) and subsequent revisions in terms of project scope, objectives, and schedules. Ensures site-specific SQAP implementation. The TM is the primary point of contact for general project problem resolution and has approving authority for the project.

1.1.2 EPA Region 10 Quality Assurance (QA) Officer

Reviews and approves the site-specific SQAP and revisions in terms of QA aspects. May conduct assessments of field activities.

1.1.3 EPA Region 10 Regional Sample Control Coordinator (RSCC)

Coordinates sample analyses performed through the EPA Contract Laboratory Program (CLP) and/or the EPA Region 10 Manchester Environmental Laboratory and provides sample identification numbers.

1.1.4 Ecology and Environment (E & E) Superfund Technical Assessment and Response Team (START) Project Manager (PM)

Provides overall coordination of field work. Provides oversight during the preparation of the site-specific SQAP. Implements the final approved version of the site-specific SQAP and records any deviations. Acts as primary contact point with the EPA TM. Receives CLP/Region 10 laboratory information from the RSCC. Acts as primary START point of contact for technical problems. Responsib-

le for the execution of decisions and courses of action deemed appropriate by the TM. In the absence of the START PM, a START Site Manager will assume the Project Manager's responsibilities.

1.1.5 E & E START QA Officer

Reviews and approves the site-specific SQAP. Conducts in-house audits of field operations. Responsible for auditing and reviewing the field activities and final deliverables, and proposing corrective action, if necessary, for nonconformities.

1.1.6 E & E START Program Manager and EPA Project Officer

Responsible for coordinating resources requested by the TM for this project and for the overall execution of the START program.

1.2 PROBLEM DEFINITION/BACKGROUND

Pursuant to EPA START Contract No. 68-W6-0008 and Technical Direction Document (TDD) No. 98-07-0011, E & E will perform a Site Inspection (SI) at the Hoy's Marine site, Newport, Oregon. The SI will consist of limited sampling at potential contaminant source and target areas for site characterization purposes. This document outlines the technical and analytical approaches E & E will employ during the Hoy's Marine SI fieldwork.

This document is a combined Field Operations Work Plan (FOWP) and site-specific Quality Assurance Project Plan (QAPP) for field sampling activities. The combined FOWP/QAPP, hereafter called the SQAP, includes a brief site summary, project objectives, sampling and analytical procedures, and QA requirements that will be used to obtain valid, representative field samples and measurements. The SQAP is intended to be combined with information presented in E & E's Quality Management Plan (QMP) for Region 10 START. Copies of the QMP and the site-specific Health and Safety Plan (prepared for the Hoy's Marine SI) are available in E & E's office located at 999 Third Avenue, Suite 1500, Seattle, Washington 98104. Standards contained in the SQAP and QMP will be used to ensure the validity of data generated by E & E for this project.

Work performed under this SQAP will be in cooperation with the Oregon Department of Environmental Quality (ODEQ) to support their site assessment program.

This section discusses the site background (Section 1.2.1), site operations and source characteristics (Section 1.2.2), and site characterization (Section 1.2.3).

1.2.1 Site Background

Information presented in this section is based on a review of site background information and interviews with property owners and representatives from various regulatory agencies.

1.2.1.1 Site Location and Description

This section describes the site location, site description, and site ownership history.

1.2.1.1.1 Site Location

Site Name:	Hoy's Marine
CERCLIS ID No.:	ORD987190840
Location:	4592 Yaquina Bay Road Newport, Oregon
Latitude:	44° 35' 45" North
Longitude:	124° 0' 43.5" West
Legal Description:	Section 22, Township 11 South, Range 11 West, Willamette Meridian, Lincoln County, Oregon
Site Owners:	Arloa J. Christiansen and Cynthia M. Steele 123 SW 12 th Street Newport, Oregon 97365 (541) 265-2340 Port of Newport 600 SE Bay Boulevard Newport, Oregon 97365 (541) 265-7758
Site Operators:	Hoy's Marine 4592 Yaquina Bay Road Newport, Oregon 97365 (541) 574-9890
Site Contacts:	Guy Hoy Hoy's Marine 4592 Yaquina Bay Road Newport, Oregon 97365 (541) 574-9890

1.2.1.1.2 Site Description

The site is located on the east bank of the Yaquina River directly north of Wiser Point (United States Geological Survey [USGS] 1984) (Figure 1-2). The city of Newport (population 8,710) is located three miles northwest of the site. The site is accessible by land via Yaquina Bay road. The site is not fenced, and can be accessed from a boat via the facility's floating dock.

Hoy's Marine is a ship building and hull refinishing business. The site comprises three adjacent tax lots, totaling approximately 0.75 acres. The site structures include a large building housing the main shop and office; a paint storage shed; a spent sandblast grit storage shed; marine ways and dry dock; and a floating dock (Figure 1-3). A graveled lot located south of the main shop was used historically for parking and miscellaneous storage, however, the lot is not currently being used by Hoy's Marine (ODEQ 1998).

The topography of the site is generally flat, but slopes steeply near the shoreline. Adjacent to the site are unused land to the northeast and southeast, the Yaquina River to the west, and developed property to the northwest which was formerly a marina, but is now vacant. The surrounding area consists of a mix of agricultural, residential, and commercial properties, as well as vacant land (ODEQ 1998). Another boat refurbishing shop, Riverbend, is located approximately 0.5 mile upstream of Hoy's Marine. Riverbend conducts sandblasting inside a machine shop (E&E 1999).

1.2.1.1.3 Site Ownership History

As mentioned previously, the site is situated on three separate parcels of property (tax lots). The land use and property ownership for all three parcels prior to 1974 is not clear. In 1974, the Port of Newport purchased one of the parcels (tax lot number 3801), and Arloa J. Christiansen and Cynthia M. Steele of Newport, Oregon jointly owned the other two parcels (tax lots 3600 and 3802). The first known commercial/industrial operation at the site was Bayside Machine Works. Bayside Machine works operated between 1974 to 1983, and leased the site properties from the Port of Newport, and Arloa J. Christiansen and Cynthia M. Steele. In 1983, the site was occupied by Fair Line Marine, who leased the property from the Port of Newport, and Arloa J. Christiansen and Cynthia M. Steele. Commercial Iron Works occupied the site between 1992 to 1993, and it is not known if Commercial Iron Works leased or rented the property. Hoy's Marine has occupied the site from 1993 to present. Presently, Hoy's Marine is in the process of purchasing tax lot 3801 from the Port of Newport. Table 1-1 summarizes the ownership and operator history of the site from 1974 to the present.

1.2.2 Site Operations and Source Characteristics

The primary operations are refurbishing vessel hulls by sandblasting old paint from ship's hulls and repainting the hulls surface (ODEQ 1998). The sandblasting and painting operations are conducted outdoors. The ships are secured in a steel cradle which runs on marine ways into the Yaquina River. Once a ship is secured in the cradle, it is pulled onto land and transferred via a perpendicular set of rails, to the dry dock area (GEM 1996). Sandblasting and painting of the vessels are conducted at the dry dock. Spot sandblasting and painting also occur within the spent grit storage shed (ODEQ 1998). Additional operations such as metal cutting by means of oxy-acetylene torches, metal grinding, and arc-welding are conducted in the main shop (GEM 1996) (Figure 1-3).

Hoy's Marine prepares surfaces by sandblasting the vessels using Kleen-Blast sandblast grit which consists of 38.1 % silicon oxide (SiO_2), 27.4 % ferric oxide (Fe_2O_3), 5.7 % aluminium oxide (Al_2O_3), and 3.9 % manganese oxide (MgO) (MSDS). The spent grit is collected and stored in the spent grit storage shed and later transported to a landfill in Corvallis, Oregon (E&E 1999).

Waste paint/thinner is stored in open containers in the paint shed where it is allowed to evaporate. Disposal procedures for the used paint and thinner containers is unknown, but will be determined during the SI fieldwork. A 300-gallon above-ground diesel storage tank is located near the southeast corner of the spent grit storage shed. An underground storage tank (UST) is located near the office portion of the main shop. The UST was reportedly closed in-place by one of the previous site operators, however, investigation of the tank in 1995 indicated that product may still remain in the tank (ODEQ 1998). Five 55-gallon drums containing motor oil and waste motor oil are located in front of the main shop. Waste oil is periodically disposed of at the Port of Newport used oil collection center (ODEQ 1998).

1.2.3 Site Characterization

This section summarizes previous site investigations (Section 1.2.3.1), discusses migration/exposure pathways and targets (Section 1.2.3.2), and describes areas of potential contamination (Section 1.2.3.3).

1.2.3.1 Site Investigations

The ODEQ has conducted a number of inspections at the Hoy's Marine site. A chronological list of previous inspections conducted by ODEQ is included in Table 1-2.

During the July 1997 preliminary assessment, ODEQ staff collected two near-surface sediment samples offshore of the site. One sample was collected below the marine way and appeared to be mostly spent grit. The second sample was collected below the tide line and north of the dry dock. The second

sample appeared to be native material (river silt). No background sample was collected. The samples were analyzed for total metals, butyltins, polychlorinated biphenyls (PCBs) and 11 polycyclic aromatic hydrocarbons (PAHs). The concentrations are summarized in [Table 1-3](#). Concentrations of the following analytes in the samples exceeded the National Oceanic and Atmospheric Administration (NOAA) Effects Range-Low (ERL) criteria for sediment: antimony, arsenic, chromium, copper, lead, nickel, zinc, total PCBs, and 10 PAHs (ODEQ 1998). Tributyltin was detected up to a concentration of 10.55 mg/kg, however, a NOAA ERL does not exist for this analyte.

In addition to the ODEQ investigations, a number of site investigations have been conducted at the site since 1989. In 1989, SRH Associates, Inc. (SRH) conducted an assessment of the site on behalf of Fair Line Marine. SRH conducted an inventory of on-site waste sources, installed exploratory soil borings around an on-site UST, and collected five composite surface soil samples in the dry dock area. SRH identified seven major concerns regarding on-site contamination. These concerns included: waste oil drums scattered throughout the facility; improper storage of paint thinner and containers; storage of numerous empty drums on-site; drums and containers of old chemicals stored on-site; storage of used petroleum distillate solvents; inadequate containment of sandblast grit; and a potentially leaking UST near the facility office (SRH 1989).

In 1995, GEM Consulting, Inc. (GEM) conducted a Level 2 site investigation for Arloa Christiansen and Cynthia Steele who own two of these lots. The intent of the investigation was to confirm the conclusions made in the 1989 SRH study. GEM excavated three test pits from which five subsurface soil samples were collected. The test pits were excavated at the UST, near the northeast corner of the main shop, and at the entrance to the paint storage shed. In addition, eight surface soil samples were collected in the paint storage shed area, the above-ground diesel tank area, a metal shavings dump area located on the northwest side of the main shop, a waste oil area on the south end of the main shop, and from spent sandblasting grit (GEM 1996).

A summary of the primary areas of concern identified by GEM is provided below (GEM 1996 and ODEQ 1998):

- **Underground Storage Tank.** According to GEM's observations, it is likely that some product still remains in the tank. One subsurface soil sample was collected from the test pit near the UST and was analyzed for total petroleum hydrocarbon (TPH) (EPA Method 418.1), TPH-G (Oregon Method TPH-G), and volatile organic compounds (VOCs) (EPA Method 8020). Two subsurface soil samples were collected from the test pit at the northeast corner of the main shop, and were analyzed for TPH, TPH-G, and VOCs. No analytes were detected above the quantitation limits in the sample collected from the test pit adjacent to the tank. VOCs and TPH were not detected in the test pit located at the northeast corner of the main shop, however, one sample contained TPH-G at a

concentration concentration of 4,100 mg/kg. This concentration exceeded the most stringent Oregon cleanup level for TPH-G of 40 mg/kg.

- **Paint Storage Shed.** Two subsurface soil samples were collected from the test pit excavated at the entrance to the paint shed, and were analyzed for VOCs (EPA Method 8240). One surface sample was collected down-slope of the shed and was analyzed for VOCs (EPA Method 8240), and total metals (EPA Method 6010). One of the test pit subsurface soil samples contained detectable concentrations of xylenes (0.030 ppm) and 1,2-dichlorobenzene (0.007 ppm) at 1.5 feet below the ground surface (bgs). Neither of these concentrations exceeded Oregon soil cleanup levels. No VOCs were detected in the samples collected at a depth of 6 inches bgs from the test pit or in the surface soil sample collected down-slope of the shed. However, the surface soil sample contained total metal concentrations of arsenic (7.4 mg/kg), cadmium (1.2 mg/kg), chromium (1,700 mg/kg), lead (180 mg/kg), and mercury (0.12 mg/kg). The concentrations of arsenic and chromium exceeded the Oregon industrial soil cleanup levels.
- **Above-Ground Diesel Tank and Waste Oil Drums.** Several 55-gallon drums containing waste oil or motor oil were stored without containment on the south side of the spent sandblast grit shed. Oil stained soils were pervasive throughout the area during GEM's inspection. One soil sample was collected from a drainage path leading from the storage area and analyzed for TPH (EPA Method 418.1) and VOCs (EPA Method 8240). The results showed a TPH-G concentration of 4,600 ppm, which exceeded the Oregon soil cleanup level. No VOCs were detected above the sample quantitation limits.
- **Spent Sandblast Grit and Paint Chips.** Spent grit and paint chips were observed throughout the shore and intertidal areas. Four samples of spent grit were collected by GEM from various locations and analyzed for total metals (EPA Method 6010). One sample was also analyzed for leachable metals following the Toxicity Characteristic Leaching Procedure (TCLP) for metals (EPA Method 1311). The results showed total metal concentrations of arsenic up to 55 mg/kg, barium up to 400 mg/kg, cadmium up to 2.4 mg/kg, chromium up to 100 mg/kg, and lead up to 310 mg/kg. The concentrations of arsenic, chromium, and lead exceeded Oregon residential cleanup levels. TCLP concentrations for all metals were below analytical detection limits.
- **Solid Waste and Metal Shavings Dump.** Previous operators of the facility disposed of metal shavings on the ground surface and in the Yaquina River near the southwest corner of the main shop. A surface soil sample was collected and analyzed for total metals (EPA Method 6010). The results showed concentrations of arsenic (37 mg/kg), barium (120 mg/kg), cadmium (11 mg/kg), chromium (1,300 mg/kg), lead (990 mg/kg), selenium (3.1 mg/kg), and silver (1.5 mg/kg). Total metals concentrations of arsenic, chromium, and lead exceeded the Oregon residential soil cleanup levels.
- **Waste Oil Area.** An above ground storage tank that contained waste oil and diesel fuel were situated on the lot south of the main shop (tax lot 3802). The tank was located on bare soil, and there was oil-like staining on the ground surface in various locations around the tank.

Two surface soil samples were collected near the main shop building to assess the presence of contamination from the tank area. The samples were analyzed for TPH (EPA Method 418.1). One of the samples was also analyzed for TCLP metals (EPA Method 1311). The samples contained TPH concentrations of 380 mg/kg and 560 mg/kg, which exceeded the Oregon soil cleanup level of 100 mg/kg. Lead was the only analyte detected in the TCLP

metals analyses at a concentration of 0.051 ppm, which is below the Oregon regulated leachate concentration for hazardous waste.

In 1997, Maul Foster & Alongi, Inc. (MFA) performed sampling at the site to support litigation proceedings between Arloa Christiansen and Hoy's Marine. Seven surface soil samples, one grit sample, and an off-site surface soil sample were collected. Four of the surface soil samples and the grit sample were analyzed for total metals (EPA Method 6010 and 7000 series). Three surface soil samples were analyzed for TPH (EPA Method 8015M). Two surface soil samples were analyzed for VOCs (EPA Method 8260) (MFA 1997).

Arsenic was detected at concentrations ranging from 25 mg/kg to 34 mg/kg; chromium was detected at concentrations ranging from 58 mg/kg to 142 mg/kg; copper was detected at concentrations ranging from 1,830 mg/kg to 2,960 mg/kg; lead was detected at concentrations ranging from 20 mg/kg to 50 mg/kg; zinc was detected at concentrations ranging from 636 mg/kg to 3,170 mg/kg; TPH (diesel range) was detected at concentrations ranging from 2,490 mg/kg to 3,400 mg/kg; TPH (heavy range) was detected at concentrations ranging from 6,240 mg/kg to 20,000 mg/kg, and naphthalene, 1,2,4-trimethylbenzene, and n-butylbenzene were detected in one sample at concentrations of 300 mg/kg, 48 mg/kg, and 68 mg/kg, respectively (MFA 1997).

Concentrations of arsenic and TPH (diesel) were reported to have exceeded Oregon soil cleanup levels (MFA 1997).

1.2.3.2 Migration/Exposure Pathways and Targets

This section discusses the groundwater migration pathway, the surface water migration pathway, the soil exposure pathway, and the air migration pathway.

1.2.3.2.1 Groundwater Migration Pathway

Hoy's Marine is underlain by well drained silty loam soils that formed in colluvium weathered from sedimentary rock. The depth to weathered bedrock in the site area is 40 to 60 inches (United States Department of Agriculture [USDA] 1997).

Groundwater is not used for drinking water within 4 miles of the site (EPA 1998a). Groundwater in the site area is reportedly brackish (E & E 1999). In approximately 1996, Hoy's Marine drilled an on-site water well intended to supply water for drinking and industrial uses at the facility. The well depth and construction details are not known, however, Mr. Hoy indicated the well was completed in bedrock. Hoy's Marine does not utilize the well because the water is brackish (E & E 1999).

1.2.3.2.1 Groundwater Migration Pathway

Stormwater from the facility is mainly sheet runoff. There is a drain in front of the main machine shop which discharges to the river. A road side ditch connects to a culvert which is buried beneath the north property line of the site. An open grate at the spent sandblast grit storage shed discharges to the buried culvert, which discharges into the river (Kretzschmar 1997). The site is located approximately 30 feet above the high water line, and as previously mentioned, the sandblasting operation is conducted on the dry dock near the water line. The total drainage area of the site is approximately 0.75 acres, which equals the area of the site. Upland areas of the site are drained by a ditch on the east side of Yaquina Bay Road, which does not cross the site.

The Yaquina River flows from the hills northeast of the site, passes the city of Toledo at approximately 14 river miles, and ultimately discharges to the Pacific Ocean via Yaquina Bay five miles downstream of the site (USGS 1984). The flow rate of the Yaquina River is 243 cubic feet per second measured at Yaquina Chitwood gaging station which is approximately 30 miles upstream of the site (USGS 1999). This station is the only flow measurement station along the whole length of the river. Hoy's Marine is located along the Yaquina River at approximately 5 river miles upstream of the river's mouth at the Yaquina Bay. The river is tidally influenced at the site; water movement in the river adjacent to the site is influenced more strongly by tidal fluctuations than by river flow (E & E 1999). Tidal influence in the river reportedly extends between 3 and 5 miles upstream of the Hoy's Marine site (ODEQ 1998). The site is located on the 100 year flood plain (National Flood Insurance Program [NFIP] 1980). The mean annual precipitation in Newport is 68.09 inches and annual snowfall is 1.1 inches (Western Regional Climate Center [WRCC] 1998). The 2-year, 24-hour rainfall average is 4.99 inches (WRCC 1998).

The Yaquina River near the site is brackish, and is not suitable for drinking (E & E 1999). Local residents draw drinking water from several nearby creeks which are tributaries of the Yaquina River (EPA 1998a). None of these creeks are hydraulically down-gradient of the site. No surface water intakes are located on the Yaquina River downstream of the site (EPA 1998a).

The Yaquina River and Yaquina Bay are used for sport fishing and other recreational and commercial activities, such as sailing, bank crabbing, and oyster farming. Swimming is not popular in the river area. In 1996, the number of fish caught by sport fishers in the Yaquina River and Yaquina Bay included 17 spring Chinook salmon, 3,603 fall Chinook salmon, and 108 winter Steelhead (Oregon Department of Fish and Wildlife [ODFW] 1998a). Coho salmon angling is no longer allowed in the Yaquina River due to its status as a federally listed threatened species (Oregon Natural Heritage Program [ONHP] 1999). Shellfish such as Dungeness crab and red rock crab are also species targeted by sport fishers in the Yaquina River and Yaquina Bay.

Commercial fishing was eliminated from the Yaquina River in 1994 to leave the river for recreational use. Commercial fishing is conducted in the Yaquina Bay area near the ocean, and is mainly

for Pacific Herring. According the ODFW, approximately 19,331 pounds of Pacific Herring were landed at Newport, Oregon in 1998. There are 19 other species with the amount of landings exceed 100,000 pounds in 1998. These species include arrowtooth flounder, mackerel, pacific ocean perch, nominal pop, rockfish, thornyhead, sablefish, chinook salmon, solefish, Albacore tuna, pacific whiting, ocean Dungeness crab, and pink shrimp (ODFW 1998b).

Several commercial oyster farms are located on the Yaquina River within one mile of the site (E & E 1999). The farms observed are upstream of Hoy's Marine (E & E 1999).

The Yaquina River is considered as a spawning and rearing area for Coho salmon (ONHP 1999), and Pacific Herring, but has not been termed critical spawning habitat (Bill Bass 1999).

One regularly flooded emergent intertidal estuarine wetland is located across the river from the site, approximately 0.3 miles away. The total length of the wetland frontage is approximately 0.1 mile (United States Department of the Interior [USDI] 1995).

Approximately 3 miles west of the site, the entire Pacific ocean coast line is designated as the Oregon Islands National Wildlife Refuge and Oregon Islands Wilderness (USGS 1984).

1.2.3.2.3 Soil Exposure Pathway

Depending on the work load, Hoy's Marine employees between 3 and 20 on-site workers. The site is readily accessible to passer-bys. No fence is installed around the property.

Table 1-4 provides population figures for people residing within one mile of the site.

1.2.3.2.4 Air Migration Pathway

Sandblasting generates significant amount of dust. In addition, the volatile organic compounds from open waste paint/thinner containers evaporate into the air. The population within 4 miles of the site is provided in **Table 1-4**.

Within 4 miles of the site, three federally listed threatened species and two federally listed endangered species have been identified. Federally listed threatened species include the western snowy plover (*Charadrius alexandrinus nivosus*), the bald eagle (*Haliaeetus leucocephalus*), and the Oregon silverspot butterfly (*Speyeria zerene hippolyta*). Federally listed endangered species include the salt-marsh bird's-beak (*Cordylanthus maritimus ssp palustris*) and the brown pelican (*Pelecaus occidentalis*) (ONHP 1999).

1.2.3.3 Areas of Potential Contamination

Sampling under the SI will be conducted at those areas considered potential contamination sources and at areas that may have been contaminated through the migration of hazardous substances from sources

on site. Based on a review of background information and discussions with site representatives, the following areas or features have been identified for inspection under the Hoy's Marine SI:

- **On-Site Contaminant Sources.** The on-site contaminant sources identified for investigation during the SI include: the former waste oil tank area south of the main shop; the former waste and iron shavings disposal area on the southwest side of the main shop; bare soil north of the main shop; the paint storage shed; the spent grit storage shed; and intertidal sediments below the dry dock. The major contaminants of concern include butyltins - tributyltin (TBT), dibutyltin (DBT), and monobutyltin (MBT) associated with marine antifouling paints from paint chips; PAHs and VOCs associated with waste oil, solvents, and thinners; and heavy metals associated with deposits of spent sandblast grit and marine paint chips.
- **Yaquina River.** The potential for adverse impacts to marine life and wetlands in the Yaquina River exists because of elevated contaminant levels detected during previous studies, particularly sediment contaminants in the Yaquina River. Both sport and commercial fishing are conducted in the river and bay area. The nearby sensitive environments include wetlands and sensitive species and habitats. As previously mentioned, the River is a spawning and rearing area critical for Coho salmon, Pacific Herring, and other species.

The sampling locations and rationale for the on-site contaminant sources and target areas are discussed in more detail in [Section 2.1.1](#).

1.3 PROJECT/TASK DESCRIPTION AND SCHEDULE

This section provides the project description ([Section 1.3.1](#)) and proposed schedule ([Section 1.3.2](#)).

1.3.1 Project Description

This section defines the objectives and scope for performing the SI activities at the Hoy's Marine site. The main goals for the Hoy's Marine SI activities are as follows:

- Collect and analyze samples to characterize the potential sources discussed in [Section 1.2.3.3](#),
- Determine off-site migration of contaminants,
- Provide the EPA with adequate information to determine whether the site is eligible for placement on the National Priorities List, and
- Document a threat or potential threat to public health or the environment posed by the site.

1.3.2 Schedule

The schedule for implementing the Hoy's Marine SI is intended to be used as a guide. Adjustments to the implementation dates and the estimated project duration may be necessary to account for variable unforeseen or unavoidable conditions that the field team may encounter. Examples include inclement weather, difficulties in accessing a sampling site, or additional time needed to complete a task. Significant schedule changes that arise in the field will be discussed with the TM at the earliest possible convenience. The proposed schedule of project work is as follows:

Activity	Start	Complete
Mobilize to Site	May 24, 1999	May 24, 1999
Sample Collection Activities	May 24, 1999	May 28, 1999
Laboratory Receipt of Samples	May 25, 1999	June 1, 1999
Demobilize from Site	May 28, 1999	May 28, 1999
Receipt of Data from Laboratory	June 25, 1999	June 25, 1999
Data Validation	July 7, 1999	July 15, 1999
Writing of Project Report	July 15, 1999	August 30, 1999
Target Project Completion Date	September 23, 1999	September 30, 1999

1.4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The project data quality objectives are to provide valid data of known and documented quality to characterize sources, determine off-site migration of contaminants, determine whether the site is eligible for placement on the National Priorities List, and document threat(s) or potential threat(s) to public health or the environment posed by the site. The data quality objective (DQO) process to be applied to this project will follow that described in the EPA document, *Guidance for the Data Quality Objectives Process* (EPA 1994a).

1.5 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

No special training requirements or certifications are required for this project except for the 40-hour Hazardous Waste Operations (HAZWOPER) class and annual refreshers. Health and safety procedures for E & E personnel are addressed in the E & E site-specific health and safety plan. As previously discussed ([Section 1.2](#)), this document is maintained in E & E's Seattle office. Included in the plan are descriptions of anticipated chemical and physical hazards, required levels of protection, health and safety monitoring requirements and action levels, personal decontamination procedures, and emergency procedures.

1.6 DOCUMENTATION AND RECORDS

This document is meant to be combined with information presented in E & E's *Quality Assurance Project Plan* (QAPP; E & E 1996a) for Region 10 START. This information is covered by the Standard Operating Procedures (SOPs) found in [Appendix A](#), the Supplemental Forms found in [Appendix B](#), and the Commercial and CLP Laboratory Statements of Work found in [Appendix D](#), and the commercial laboratory Quality Assurance Manual, which has been previously reviewed by E & E. A copy of the START QAPP is available in E & E's Seattle office. Standards contained in the SOPs, the START QAPP, and the QMP will be used to ensure the validity of data generated by E & E for this project.

Following the completion of fieldwork and the receipt of analytical data, a report summarizing project findings will be prepared. Project files including work plans, reports, analytical data packages, correspondence, chain-of-custody documentation, logbooks, corrective action forms, referenced materials, and photographs will be provided to the EPA TM at the close of the project. Further, a CD-Rom deliverable containing the SI report, will be provided.

Table 1-1 OWNERSHIP AND OPERATOR HISTORY HOY'S MARINE SITE INSPECTION NEWPORT, OREGON			
TAX LOT NUMBER	DATE	OWNER	OPERATOR
3600 and 3802	1974-1983	Arloa Christiansen and Cynthia Steele	Bayside Machine Works
	1983-1992		Fair Line Marine
	1992-1993		Commercial Iron Works
	1993-present		Hoy's Marine
3801	1974-1983	Port of Newport	Bayside Machine Works
	1983-1992		Fair Line Marine
	1992-1993		Commercial Iron Works
	1993-present		Hoy's Marine

Table 1-2 LIST OF PREVIOUS INVESTIGATIONS AT THE SITE BY ODEQ HOYS MARINE SITE INSPECTION NEWPORT, OREGON		
Date	Nature of Inspection	Major Areas of Concern
May 6, 1997 (Perry 1997)	Water quality complaint	Stormwater runoff from the site; wastewater discharges from washing activities.
July 25, 1997 (Kretzschmar 1997)	Preliminary Assessment	Wastewater discharge from washing activities; sandblasting grit disposal into the river; painting and paint shed; fuel tanks; open burning of wastes; stormwater runoff.
September 11, 1997 (Parr 1997)	Compliance with the ODEQ and Federal Hazardous Waste Rules	Waste streams include wastewater and spent sandblasting grit; illegal treatment of waste thinner/paint; fuel tanks and used oil tanks; spillage of chemicals on the ground.

Reference: ODEQ 1998

Table 1-3 1997 ODEQ SEDIMENT SAMPLE ANALYTICAL RESULTS (mg/kg) HOY'S MARINE SITE INSPECTION NEWPORT, OREGON				
	Samples		Standards	
	Beneath Haul-out	North of Haul-out	NOAA ER-L ¹	Washington ²
Antimony	14	7.7	2	NA
Chromium	148	140	80	260
Zinc	2070	690	120	410
Lead	167	53.9	35	450
Nickel	151	45.1	30	NA
Copper	2420	901	70	390
Arsenic	70.6	23.1	33	57
PCBs	0.5653	0.4051	0.05	NA
Butyltins (total)	6.8117	14.0714	NA	NA
Tributyltin	4.15	10.55	NA	0.030
PAHs above NOAA ER-L ¹	10	8		

Reference: ODEQ 1998

¹ NOAA, Effective Level Criteria.

² Washington Sediment Management Standards, April 1991, Chapters 173-204 WAC.

Key:

mg/kg = milligram per kilogram.

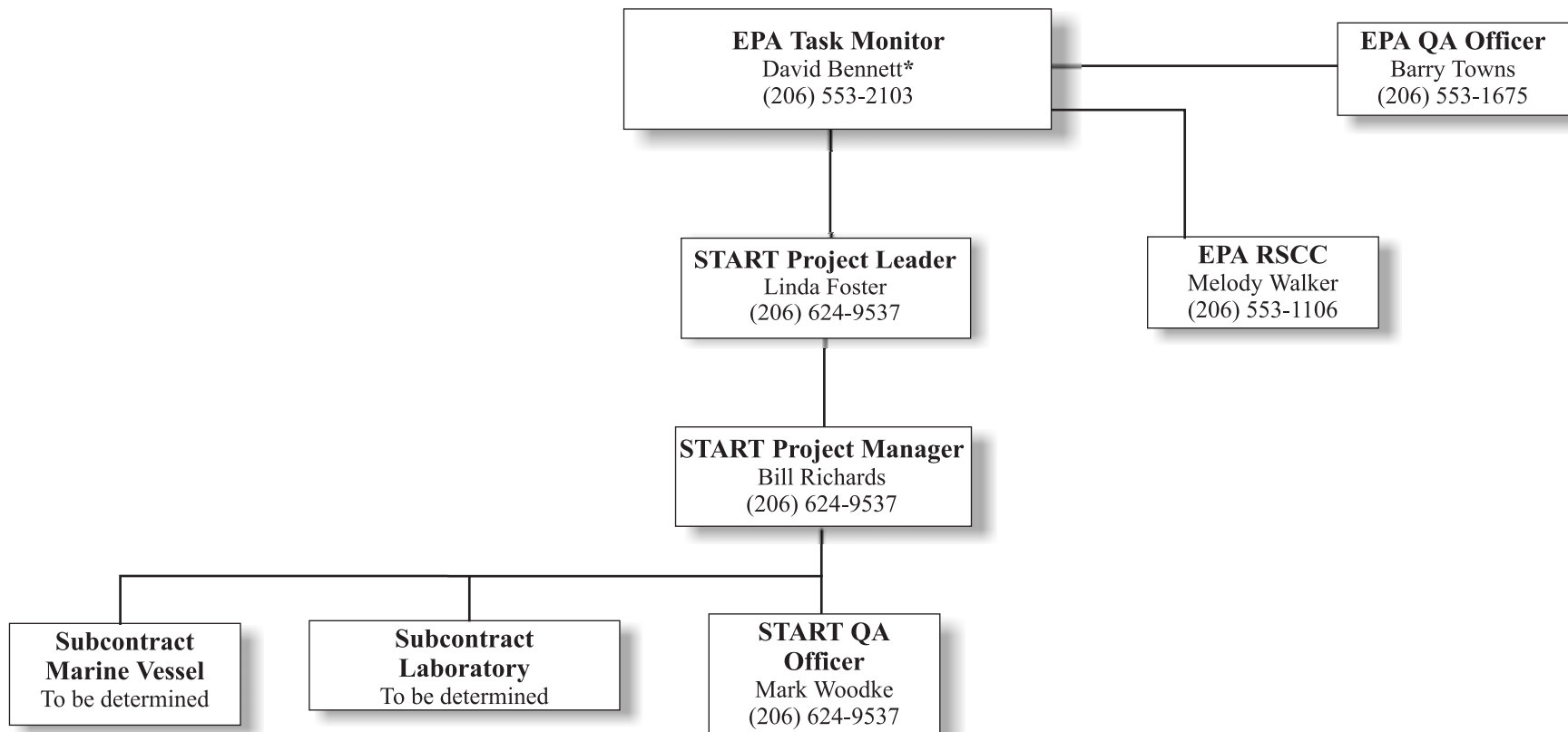
NA = Not Available.

PAHs = Polycyclic Aromatic Hydrocarbons.

PCBs = Polychlorinated Biphenyls.

<p>Table 1-4</p> <p>POPULATIONS AND WETLAND ACREAGE WITHIN A 4-MILE RADIUS</p> <p>HOY'S MARINE SITE INSPECTION</p> <p>NEWPORT, OREGON</p>		
DISTANCE (MILES)	RESIDENTS	WETLAND ACREAGE
On a source	3	0
0 to 1/8	1	0
1/8 to 1/4	6	2.0
1/4 to 1/2	24	6.0
1/2 to 1	94	0
1 to 2	407	220.0
2 to 3	2001	220.0
3 to 4	7874	20.0
Total	10,410	468.0

Reference: EPA 1998a



KEY: _____
 * Approving Authority



Source: USGS, Newport South, and Newport North, Oregon, 1984



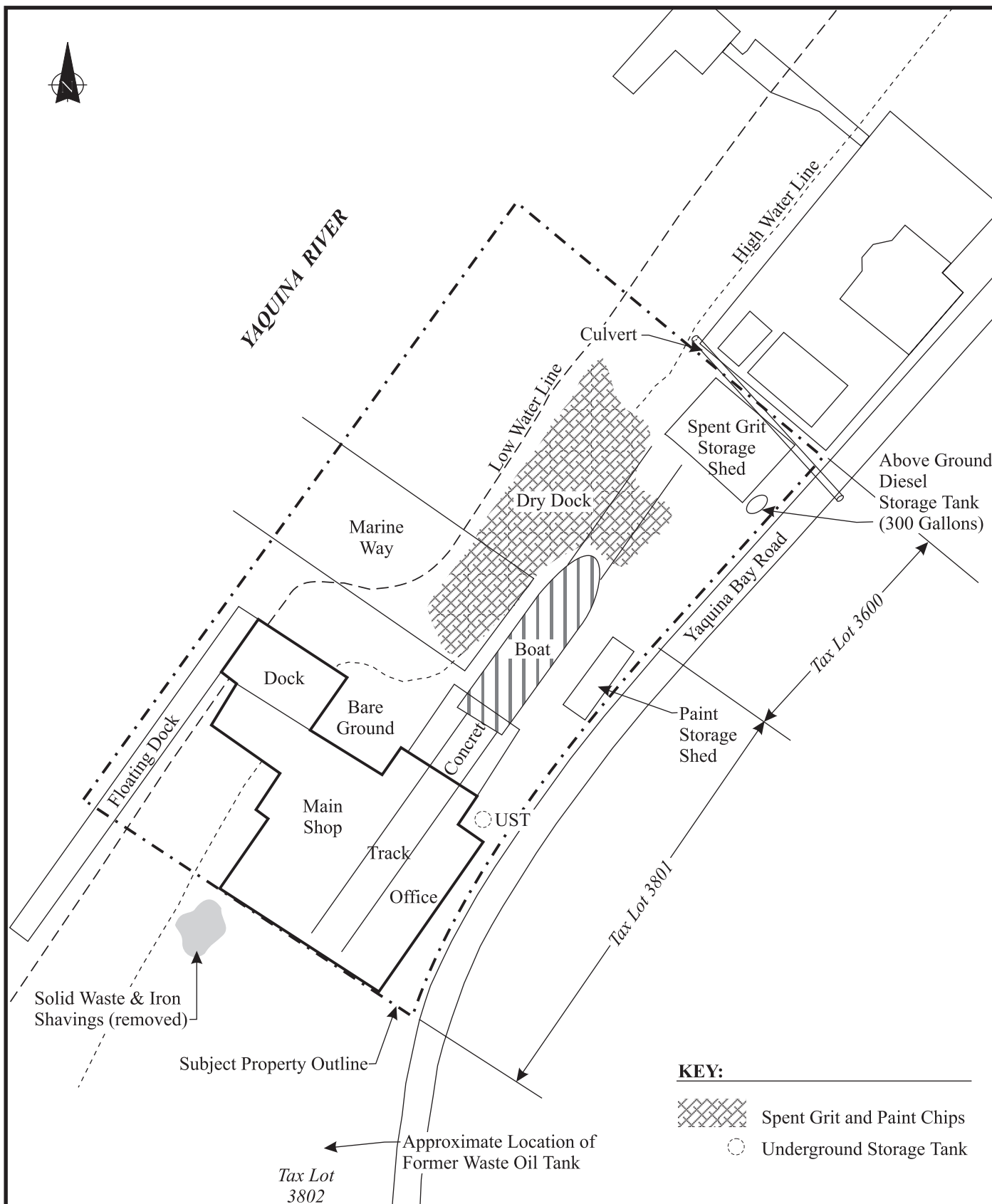
ecology and environment, inc.
International Specialists in the Environment
Seattle, Washington

HOY'S MARINE SITE Newport, Oregon



Figure 1-2
SITE VICINITY MAP

Drawn: AES	DATE: 4/19/99	JOB NO. CG1101SIT0	Dwg.No. CG1101 1-2
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ecology and environment, inc.
International Specialists in the Environment
Seattle, Washington

HOY'S MARINE SITE
Newport, Oregon

Not to Scale

Drawn:
AES

DATE:
4/19/99

JOB NO.
CG1101SIT0

Dwg.No.
CG1101 1-3

Figure 1-3
SITE LOCATION MAP

2. MEASUREMENT/DATA ACQUISITION

2.1 SAMPLING PROCESS DESIGN (EXPERIMENTAL DESIGN)

During the Hoy's Marine SI, samples will be collected from locations or features considered potential contamination sources, from selected potential hazardous substance migration pathways, and from potential targets in those pathways. The locations or features to be sampled have been determined based on information derived from a review of background information and interviews with site representatives. **Table 2-1** provides sample information regarding the sampling design and whether the measurement is considered critical or non-critical.

At the time of sampling, site-specific conditions (i.e., topography or visual evidence of contamination) will be evaluated and incorporated, when applicable, into the placement of sampling locations. Other conditions potentially contributing to deviations from the projected sampling locations include new observations or information obtained in the field that warrant an altered sampling approach, difficulty in reaching a desired soil sampling depth caused by adverse soil conditions or obstructions, or limited access to a sampling location. Significant deviations from the planned sampling locations or number of samples to be collected will be discussed with the EPA TM before implementation and will be documented on a Sample Plan Alteration form. A sample form is included in **Appendix B**. Every attempt will be made to collect representative samples with the equipment being used.

This section will describe sample locations (**Section 2.1.1**); the global positioning system (GPS) (**Section 2.1.2**); logistics (**Section 2.1.3**); cooler return (**Section 2.1.4**); coordination with Federal, State, and local authorities (**Section 2.1.5**); and the proposed schedule (**Section 2.1.6**).

2.1.1 Sample Locations

Sample locations have been selected to achieve the objectives discussed in **Section 1.3.1**. All samples will be submitted for off-site fixed laboratory analysis for target analyte list (TAL) metals (Contract Laboratory Program Analytical Service [CLPAS] ILM04.0). In addition, soil samples collected from associated stained soil areas will be analyzed for VOCs (including BTEX; CLPAS OLM03.2) and semi-volatile organic compounds (SVOCs) (CLPAS OLM03.2) using CLP standards. Analysis for TBT, which also includes analyses for dibutyltin and monobutyltin, using a published method by Stallard et al. (Stallard 1988) and total organic carbon (TOC) using EPA SW-846 Method 9060 in sediments will be

conducted by a commercial laboratory contracted by START. The sediment samples will also be analyzed for grain size in the laboratory. Proposed sample locations are illustrated on **Figures 2-1 and 2-2**.

Table 2-2 presents the anticipated number and types of samples, analytical methods, specific requirements for sample container size and type, sample preservation and holding times, and special handling requirements for samples expected to be collected at the site. **Table 2-3** summarizes the number of QA/quality control (QC) samples to be submitted according to the method requirements. A summary of sampling locations and rationale is provided below:

- **Former Waste Oil Tank Area.** The area may be contaminated from storage of waste oil tanks. Two surface soil samples will be collected from the former waste oil tank area. These samples will be used to confirm a release of hazardous substances to surface soil and to document waste characteristics.
- **Former Solid Waste and Iron Shavings Dump Area.** The area may be contaminated from solid waste and iron shavings dumping. One surface soil sample will be collected from the soil waste dump area, and one surface soil sample will be collected from behind the main machine shop where iron shavings were deposited. These samples will be used to identify the presence of hazardous substances in surface soil on-site and to document waste characteristics.
- **Bare Ground in Front of the Main Shop.** The area may be contaminated from the oil residue washed out from the concrete floor in front of the main shop. Two surface soil samples will be collected from the bare ground in front of the shop. These samples will be used to confirm a release of hazardous substances to surface soil and to document waste characteristics.
- **Paint Storage Shed.** Paint and solvent stains were observed during the START site visit on the floor of the paint storage shed. Since no containment is installed in the paint storage shed, storm water runoff may wash the paint residue down to the Yaquina River. Two surface soil samples will be collected from the entrance of the paint storage shed, and two surface soil samples will be collected from the water-facing side of the shed. These samples will be used to confirm a release of hazardous substances to surface soil and to document waste characteristics.
- **Between the Paint Storage Shed and Dry Dock.** The storm water runoff route may be contaminated from the storm water runoff from the paint storage shed contaminants since no containment is installed. One surface soil sample will be collected between the paint storage shed and the dry dock. This sample will be used to document migration of hazardous substances attributable to the paint storage shed toward the Yaquina River.
- **Spent Grit Storage Shed.** An above ground diesel tank is, and waste oil drums were, located at the southeast corner of the spent grit storage shed. Contamination to surface soil is possible. One surface soil sample from the entrance, and one from the south side, of the spent grit storage shed, will be collected. One sample will be collected from the spent grit pile in the storage shed. These samples will be used to confirm a release of hazardous substances to surface soil and to document waste characteristics.

- **Yaquina River Intertidal Surface Sediments Beneath Dry Dock.** The area beneath the dry dock is believed to be the main probable point of entry (PPE) for the surface water pathway. Four intertidal surface samples will be collected from beneath the dry dock. These samples will be used to document a release of hazardous substances to surface water.
- **Yaquina River Sub-Tidal Sediments.** Two transects containing a total of 12 subtidal surface sediment samples will be collected. One transect will be located at 20 feet from the shoreline and the other transect will be located at approximately 50 feet from the shoreline. Both transects contain six samples at an interval of 40 feet. These samples will be used to document the extent of a release of hazardous substances to surface water.
- **Yaquina River Sub-Tidal Sediments Between Riverbend Site and Hoy's Marine Site.** The Riverbend site, conducting a similar operation to Hoy's Marine, is located upstream of the Hoy's Marine site. Two transects containing a total of 8 subtidal surface sediment samples will be collected between Riverbend site and Hoy's Marine site to determine the source of hazardous substance contamination in sediments off-shore of Hoy's Marine. One transect of 4 samples will be located at 20 feet from the shoreline and the other transect of 4 samples will be located at 50 feet from the shoreline. The samples are intended to delineate a gradient of contamination between the Riverbend and Hoy's Marine sites. In addition, the samples will be collected at similar depths and in similar sedimentary regimes. A visual grain-size analysis will be conducted on all sediment samples to help ensure adequate comparability of sample results. These samples will be used to confirm a release of hazardous substance attributable to Hoy's Marine site.
- **Yaquina River Sub-Tidal Sediments Downstream of Hoy's Marine.** A number of commercial and industrial operations are located within 1 mile downstream of the Hoy's Marine site. Two transects containing a total of 4 sub-tidal surface sediment samples will be collected downstream of Hoy's Marine to determine the source of hazardous substance contamination in sediments downstream of Hoy's Marine. The samples will be located approximately 1 mile downstream of Hoy's Marine site. One transect of 2 samples will be located at 20 feet from the shoreline and the other transect of 2 samples will be located at 50 feet from the shoreline. The samples will be collected at similar depths and in similar sedimentary regimes. A visual grain-size analysis will be conducted on all sediment samples to help ensure adequate comparability of sample results. These samples will be used to confirm a release of hazardous substance attributable to Hoy's Marine site.
- **Off-Shore Wetland Sediments.** The wetland may be impacted by the migration of hazardous substances from Hoy's Marine. Three intertidal surface sediment samples will be collected along the frontage of the wetland, at intervals of approximately 550 feet. These samples will be used to determine target exposure to hazardous substances.
- **Background Samples.** Two representative background surface soil samples having similar characteristics to the soil at Hoy's Marine site will be collected. Three background surface sediment samples will be collected at least 5 miles upstream of the Hoy's Marine site. These samples include one intertidal surface sediment sample collected at the shoreline, and two sub-tidal surface sediment samples collected at approximately 20 feet and 50 feet, respectively, from the shoreline. The samples will be collected at similar depths and in similar sedimentary regimes. A visual grain-size analysis will be conducted on all sediment samples to ensure adequate comparability of sample results.

2.1.2 Global Positioning System

GPS units with data loggers will be used to identify the location coordinates of every sample collected, as well as to delineate the boundaries of the potential source areas. GPS coordinates will be provided in the final SI report as an appendix. The START will obtain differential correction data from a local source prior to the start of the survey in order to improve the survey resolution.

2.1.3 Logistics

Hoy's Marine is accessible by Yaquina Bay Road. All property owners will be informed and consent letters to access their properties for SI activities will be sent to the owners three weeks prior to the planned initiation date of field activities.

Sample aliquots collected for CLP laboratory analysis will be delivered to the EPA Region 10 laboratory or an alternative laboratory as directed by the EPA. Sample aliquots collected for analysis of TOC and TBT will be transported to a commercial laboratory under contract to the START. All samples will be shipped at the end of the fieldwork by commercial airlines for express delivery. Sample control and shipping are discussed in [Section 2.3](#).

A subcontracted boat will be used for the collection of sub-tidal sediment samples. The boat will meet all requisite United States Coast Guard safety standards, and will have the necessary equipment for the retrieval and handling of collected sediment samples.

2.1.4 Cooler Return

For laboratories other than the EPA Region 10 laboratory, E & E will provide a completed air bill accompanied by a plastic envelope with adhesive back and an address label in the chain-of-custody bags taped to the lid inside coolers so the laboratory can return the coolers to E & E. The air bill will contain the following notation: "Transportation is for the U.S. Environmental Protection Agency and the total actual transportation charges paid to the carrier(s) by the consignor or consignee shall be reimbursed by the Government, pursuant to cost reimbursement contract No. 68-W6-0008." This notation will enable the laboratories to return the sample coolers to E & E's warehouse. The air bills will be marked for second-day economy service and will contain the appropriate TDD number for shipment. For the EPA Region 10 laboratory, an arrangement by E & E for cooler return in this manner is not required.

2.1.5 Coordination with Federal, State, and Local Authorities

The START will keep the TM apprised of field event progress and issues that may affect the schedule or outcome of the SI, discuss problems encountered, inform the EPA of unusual contacts with the public or the media, and obtain guidance from the EPA regarding project activities when required.

Additionally, the START will notify the EPA RSCC with changes to the sampling schedule and provide shipping information on every sample shipment within 24 hours of shipment or before noon on Friday for Saturday delivery.

Before initiation of the SI field activities, the START will provide notification to Mr. Steve Nichols of ODEQ, Guy Hoy of Hoy's Marine (property operator), Don Mann of the Port of Newport (property owner), and Arloa J. Christiansen (property owner).

2.1.6 Schedule

The schedule for implementing the Hoy's Marine SI is intended to be used as a guide. Adjustments to the implementation dates and the estimated project duration may be necessary to account for variable unforeseen or unavoidable conditions that the field team may encounter. Examples include inclement weather, difficulties in accessing a sampling site, or additional time needed to complete a task. Significant schedule changes that arise in the field will be discussed with the TM at the earliest possible convenience.

The START is targeting May 24, 1999, as the earliest period to conduct the SI fieldwork, which is estimated to take 5 days, including travel time to and from the site. This period comprises one day of mobilization and one day of demobilization, and four days to complete field activities. Work will be conducted during daylight hours only.

2.2 SAMPLING METHODS REQUIREMENTS

This section describes sampling methodologies (Section 2.2.1); sampling equipment decontamination (Section 2.2.2); investigation-derived wastes (Section 2.2.3); and standard operating procedures (SOPs) (Section 2.2.4).

2.2.1 Sampling Methodologies

The START PM and EPA TM will be responsible for ensuring that appropriate sample collection procedures are followed and will take appropriate actions to correct the deficiencies. All samples collected will be maintained under chain-of-custody and will be stored and shipped in iced coolers.

- **Surface Soil Sampling.** Surface soil (0 to 6 inches bgs) will be collected using a dedicated Stainless steel spoon or trowel. Collected material for non-VOC analysis will be placed in a dedicated Stainless steel bowl, homogenized thoroughly and placed into pre-labeled sample containers. Soil sample aliquots intended for VOC analysis will be placed directly into pre-labeled sample containers. Guidelines in the soil sampling SOP in [Appendix A](#) will be followed.

- **Intertidal Bulk Sediment Sampling.** Intertidal bulk sediment samples will be collected from the top 6 inches by using a dedicated stainless steel spoon or trowel. The collected sediments will be homogenized thoroughly in a dedicated stainless steel bowl, decanting excess water before they are placed into pre-labeled sample containers. Guidelines in the sediment sampling SOP in [Appendix A](#) will be followed. Intertidal sediment samples will be collected below the mean water river water line.
- **Sub-Tidal Bulk Sediment Sampling.** Sub-tidal bulk sediment samples will be collected from the top 6 inches using a non-dedicated stainless steel van Veen bottom grab sampler. The sample portion will be extracted from material not in contact with the sampler using a dedicated stainless steel spoon. Use of the sampler, and the sediment sampling process will follow Puget Sound benthic sampling protocols specified in guidance prepared for EPA Region 10 (PTI 1990). For all sediment samples collected, the samples will be homogenized thoroughly in a dedicated stainless steel bowl, decanting excess water before they are placed into pre-labeled sample containers. All samples will be collected at a similar tidal cycle. Guidelines in the sediment sampling SOP in [Appendix A](#) will be followed.

2.2.2 Sampling Equipment Decontamination

To the greatest extent possible, disposable and/or dedicated personal protective and sampling equipment will be used to avoid cross-contamination. All sampling equipment to be used for the collection of on-site soil and sediment samples will be dedicated, and therefore will not require decontamination. When required, decontamination will be conducted in a central location, upwind and away from suspected contaminant sources. The decontamination procedures for non-dedicated stainless steel sampling equipment will consist of the following steps:

- Scrub to remove all visible material,
- Scrub with a brush using a non-phosphate detergent and potable water,
- Rinse with potable water,
- Rinse with diluted nitric acid,
- Rinse with deionized water, and
- Air dry.

2.2.3 Investigation-Derived Wastes

START field team members will make every effort to minimize the generation of investigation-derived wastes throughout the field event. Attempts will be made to evaporate wastewater from decontamination operations on site. Any wastewater that cannot be evaporated will be contained in 55-gallon drums, labeled, and disposed of at an approved facility. Based on SI analytical results from soil and sediment samples, disposable personal protective clothing and sampling equipment generated during

field activities will be rendered unusable by tearing (when appropriate), bagged in opaque plastic garbage bags and disposed of at a municipal landfill.

2.2.4 Standard Operating Procedures

START will utilize the following SOPs (see [Appendix A](#)) while performing field activities:

- Field Activities Logbook;
- Soil Sampling;
- Sediment Sampling;
- Sample Packaging and Shipping;
- Site Entry Procedures; and
- Sampling Equipment Decontamination.

2.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

This section describes sample identification and chain-of-custody procedures that will be used for the Hoy's Marine SI field activities. The purpose of these procedures is to ensure that the quality of samples is maintained during collection, transportation, storage, and analysis. All chain-of-custody requirements comply with E & E's SOPs for sample handling. All sample control and chain-of-custody procedures will follow the EPA's *User's Guide to the Contract Laboratory Program* (EPA, 1991).

Examples of sample documents used for custody purposes are provided in [Appendix C](#) and include the following:

- Sample identification numbers,
- Sample tags or labels,
- Custody seals,
- Chain-of-custody and traffic report records,
- Field logbooks,
- Sample collection forms,
- Analytical request forms, and
- Analytical records.

During the field effort, the site manager or delegate is responsible for maintaining an inventory of these sample documents. This inventory will be recorded in a cross-referenced matrix of the following:

- Sample location,
- Sample identification number,
- Analyses requested and request form number(s),
- Chain-of-custody record numbers,
- Bottle lot numbers, and
- Air bill numbers.

Brief descriptions of the major sample identification and documentation records and forms are provided below.

2.3.1 Sample Identification

All samples will be identified using the sample numbers assigned by the EPA Regional Sample Coordination Center. Each sample label will be affixed to the jar and covered with Mylar tape. A sample tracking record will be kept as each sample is collected. The following will be recorded: location, matrix, sample number, observations, and depth. In addition to the EPA-assigned sample number, samples will be tracked with a sample code system designed to allow easy reference to the sample's origin and type. The sample code key will not be provided to the laboratory. **Table 2-4** summarizes the sample tracking code and location.

2.3.1.1 Sample Tags and Labels

Sample tags attached to or fixed around the sample container will be used to identify all samples collected in the field. The sample tags will be placed on bottles so as not to obscure any QA/QC lot numbers on the bottles, and sample information will be printed legibly. Field identification will be sufficient to enable cross-reference with the project logbook. For chain-of-custody purposes, all QA/QC samples will be subject to the same custodial procedures and documentation as site samples.

To minimize handling of sample containers, labels will be completed before sample collection to the extent possible. In the field the label will be filled out completely using waterproof ink, then attached firmly to the sample containers and protected with Mylar tape. The sample label will provide the following information:

- Sample number,
- Sample location number,
- Date and time of collection,

- Analysis required, and
- pH and preservation (when applicable).

2.3.1.2 Custody Seals

Custody seals are preprinted gel-type seals, designed to break into small pieces if the seals are disturbed. Sample shipping containers (e.g., coolers, drums, and cardboard boxes, etc., as appropriate) will be sealed in as many places as necessary to ensure security. Seals will be signed and dated before use. Clear tape will be placed over the seals to ensure that seals are not broken accidentally during shipment. Upon receipt at the laboratory, the custodian will check (and certify by completing the package receipt log) that seals on shipping containers are intact.

2.3.1.3 Chain-of-Custody Records and Traffic Reports

For samples to be analyzed at a CLP laboratory, the chain-of-custody record and analytical traffic report forms will be completed as described in the *CLP User's Guide*. The chain-of-custody record and analytical traffic reports will be completed fully at least in duplicate by the field technician designated by the site manager as responsible for sample shipment to the appropriate laboratory. Information specified on the chain-of-custody record will contain the same level of detail found in the site logbook, except that the on-site measurement data will not be recorded. The custody record will include the following information:

- Name and company or organization of person collecting the samples;
- Date samples were collected;
- Type of sampling conducted (composite or grab);
- Sample number (using those assigned by the EPA RSCC);
- Location of sampling station (using the sample code system described in [Table 2-4](#));
- Number and type of containers shipped;
- Analysis requested; and
- Signature of the person relinquishing samples to the transporter, with the date and time of transfer noted and signature of the designated sample custodian at the receiving facility.

If samples require rapid laboratory turnaround, the person completing the chain-of-custody record will note these or similar constraints in the remarks section of the custody record.

The relinquishing individual will record all shipping data (e.g., air bill number, organization, time, and date) on the original custody record, which will be transported with the samples to the laboratory and

retained in the laboratory's file. Original and duplicate custody records, together with the air bill or delivery note, constitute a complete custody record. It is the site manager's responsibility to ensure that all records are consistent and that they become part of the permanent job file.

2.3.1.4 Field Logbooks and Data Forms

Field logbooks (or daily logs) and data forms are necessary to document daily activities and observations. Documentation will be sufficient to enable participants to reconstruct events that occurred during the project accurately and objectively at a later time. All daily logs will be kept in a bound notebook containing numbered pages. All entries will be made in waterproof ink, dated, and signed. No pages will be removed for any reason.

Minimum logbook content requirements are described in the E & E SOP entitled *Preparation of Field Activities—Logbooks*. If corrections are necessary, these corrections will be made by drawing a single line through the original entry (so that the original entry is legible) and writing the corrected entry alongside. The correction will be initialed and dated. Corrected errors may require a footnote explaining the correction.

2.3.1.5 Photographs

Photographs will be taken as directed by the team leader. Documentation of a photograph is crucial to its validity as a representation of an existing situation. The following information will be noted in the project or task log concerning photographs:

- Date, time, and location where photograph was taken;
- Photographer (signature);
- Weather conditions;
- Description of photograph taken;
- Reasons why photograph was taken;
- Sequential number of the photograph and the film roll number;
- Camera lens system used; and
- Direction.

2.3.2 Custody Procedures

The primary objective of chain-of-custody procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from collection to completion of all required analyses. A sample is in custody when it is:

- In someone's physical possession,
- In someone's view,
- Locked up, or
- Kept in a secured area that is restricted to authorized personnel.

2.3.2.1 Field Custody Procedures

The following guidance will be used to ensure proper control of samples while in the field:

- As few people as possible will handle samples.
- Coolers or boxes containing cleaned bottles will be sealed with a custody tape seal during transport to the field or while in storage before use. Sample bottles from unsealed coolers or boxes, or bottles that appear to have been tampered with, will not be used.
- The sample collector will be responsible for the care and custody of collected samples until they are transferred to another person or dispatched properly under chain-of-custody rules.
- The sample collector will record sample data in the field logbook.
- The site team leader will determine whether proper custody procedures were followed during the fieldwork and will decide if additional samples are required.

When transferring custody (i.e., releasing samples to a shipping agent), the following will apply:

- The coolers in which the samples are packed will be sealed and accompanied by two chain-of-custody records. When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on the chain-of-custody record. This record will document sample custody transfer.
- Samples will be dispatched to the laboratory for analysis with separate chain-of-custody records accompanying each shipment. Shipping containers will be sealed with custody seals for shipment to the laboratory. The chain-of-custody records will be signed by the relinquishing individual, and the method of shipment, name of courier, and other pertinent information will be entered in the chain-of-custody record before placement in the shipping container.
- All shipments will be accompanied by chain-of-custody records identifying their contents. The original record will accompany the shipment. The other copies will be distributed appropriately to the site team leader and site manager.

- If sent by common carrier, a bill of lading will be used. Freight bills and bills of lading will be retained as part of the permanent documentation.

2.3.2.2 Laboratory Custody Procedures

A designated sample custodian at the laboratory will accept custody of the shipped samples from the carrier and enter preliminary information about the package into a package or sample receipt log, including the initials of the person delivering the package and the status of the custody seals on the coolers (i.e., broken versus unbroken). The custodian responsible for sample log-in will follow the laboratory's SOP for opening the package, checking the contents, and verifying that the information on the chain-of-custody agrees with samples received. The commercial laboratory will follow its internal chain-of-custody procedures as stated in the laboratory QA Manual.

2.4 ANALYTICAL METHODS REQUIREMENTS

This section discusses the analytical strategy (Section 2.4.1) and the analytical methods (Section 2.4.2).

2.4.1 Analytical Strategy

Analysis of samples collected during the SI will be performed by several possible means. The EPA Region 10 laboratory (or alternative laboratory designated by the EPA) will perform all requested analyses with the exception of sample aliquots for TOC, and TBT which will be analyzed by a commercial laboratory under subcontract to the START.

The analyses to be applied to samples sent to the laboratory are listed in Table 2-2. These analyses were selected based on the probable hazardous substances used or potentially released to the environment, given the known or suspected site usage.

2.4.2 Analytical Methods

Samples designated for off-site analytical laboratory analyses will be submitted to the EPA Region 10 laboratory located in Manchester, Washington, or an alternative laboratory designated by the EPA, and the START-subcontracted commercial laboratory. Analyses will take place under the standard 8-week turnaround time period. Table 2-3 summarizes laboratory instrumentation and methods to be used for the SI.

All instruments and equipment used during field and fixed laboratory sample analyses will be operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations, as well as criteria set forth in the applicable analytical methodology references.

In the cases where laboratory results exceed QC acceptance criteria, reextraction and/or reanalysis will occur as indicated in the applicable analytical method. Commercial laboratory results (preliminary data) will be generally be available within 2 weeks of sample receipt. The respective laboratory analysts will be responsible to ensure that appropriate sample analysis procedures are followed and take appropriate actions to ensure deficiency correction. A copy of the Statement of Work for the commercial laboratory analyses, including copies of the applicable analytical methods, will included in **Appendix D** after a subcontract laboratory for this work has been secured.

2.5 QUALITY CONTROL REQUIREMENTS

QC checks for sample collection will be accomplished by a combination of chain-of-custody protocols and laboratory quality assurance as prescribed in the sampling or analytical methods. No QC samples (i.e. double blind performance evaluation samples) are planned for this activity outside of normal laboratory QC criteria outlined in the analytical methods. In accordance with the objectives outlined in this document and the QA Levels defined by the EPA (EPA 1993), the EPA has defined the data quality objectives and has determined that the sampling and analyses performed under this sampling effort will conform to the definitive data without error and bias determination criteria.

2.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

The field equipment used during this project includes the GPS unit. Testing, inspection, and maintenance of this instrument will be performed in accordance with the manufacturer's recommendations. Spare parts for the field equipment will be available from the manufacturer generally within 24 hours.

2.7 INSTRUMENT CALIBRATION AND FREQUENCY

All instruments and equipment used during fixed laboratory sample analyses will be operated, calibrated, and maintained according to the manufacturer's guidelines and recommendations, as well as criteria set forth in the applicable analytical methodology references and/or in accordance with the laboratory's QA and SOPs.

For the field instrumentation (GPS unit), the calibration will be performed in accordance with the manufacturer's recommendations.

2.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

This information is covered by the SOPs, the START QAPP, and the START QMP (E & E 1996c). Standards contained in these documents will be used to ensure the validity of data generated by E & E for this project.

2.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

No data will be used from other sources.

2.10 DATA MANAGEMENT

This document is meant to be combined with information presented in E & E's QAPP and QMP for Region 10 START. Copies of the START QAPP and QMP are available in E & E's Seattle office. Standards contained in these documents will be used to ensure the validity of data generated by E & E for this project.

Table 2-1

**SAMPLE INFORMATION SUMMARY
HOY'S MARINE SITE INSPECTION
NEWPORT, OREGON**

Project Sampling Location	Para- meter	Design Rationale	Sampling Design Assumptions	Sample Selection Procedures	Measurement Classification (Critical/ Noncritical)	Nonstandard Method Validation
Former Waste Oil Tank Area Surface Soil	SVOCs Metals	Confirm a Release of Hazardous Substances	Contaminated Surface Soil Mixed with Metal Based Spent Grit	Two Representative Samples will be Selected	Critical	(*)
Former Solid Waste & Iron Shavings Dump Area Surface Soil	SVOCs Metals	Identify Presence of Hazardous Substances	Contaminated Surface Soil Mixed with Metal Based Spent Grit	Two Representative Samples will be Selected	Critical	(*)
Bare Ground in Front of the Main Shop Surface Soil	SVOCs VOCs PCBs Metals	Confirm a Release of Hazardous Substances	Contaminated Surface Soil Mixed with Metal Based Spent Grit	Two Representative Samples will be Selected	Critical	(*)
Paint Storage Shed Surface Soil	VOCs SVOCs PCBs Metals	Confirm a Release of Hazardous Substances	Contaminated Surface Soil Mixed with Metal Based Spent Grit	Four Representative Samples will be Selected	Critical	(*)
Between the Paint Storage Shed and Dry Dock	VOCs SVOCs PCBs Metals	Confirm Migration of Hazardous Substances	Contaminated Surface Soil Mixed with Metal Based Spent Grit	One Representative Samples will be Selected	Critical	(*)
Spent Grit Storage Shed Surface Soil and Spent Grit	SVOCs Metals	Confirm a Release of Hazardous Substances	Contaminated Surface Soil Mixed with Metal Based Spent Grit	Two Representative Surface Soil Samples, One Spent Grit Sample will be Selected	Critical	(*)
Beneath Dry Dock Inter- Tidal Surface Sediments	SVOCs VOCs TOC PCBs Metals TBT	Confirm a Release of Hazardous Substances to the river.	Contaminated Intertidal Sediments Mixed with Metal Based Spent Grit	Four Representative Samples will be Selected	Critical	(*)

<p align="center">Table 2-1</p> <p align="center">SAMPLE INFORMATION SUMMARY</p> <p align="center">HOY'S MARINE SITE INSPECTION</p> <p align="center">NEWPORT, OREGON</p>						
Project Sampling Location	Parameter	Design Rationale	Sampling Design Assumptions	Sample Selection Procedures	Measurement Classification (Critical/ Noncritical)	Nonstandard Method Validation
Yaquina River at Hoy's Marine Subtidal Surface Sediments	SVOCs TOC PCBs Metals TBT	Confirm a release of Hazardous Substances to the river.	Contaminated Surface Sediments	Twelve Samples in Two Transects at Six Samples per Transect will be Selected at 20 and 50 feet to the Shoreline.	Critical	(*)
Yaquina River between Riverbend and Hoys Marine Subtidal Surface Sediments	SVOCs TOC PCBs Metals TBT	Confirm a Release of Hazardous Substances Attributable to Hoys Marine Site	Contaminated Surface Sediments	Eight Samples in Two Transects at Four Samples per Transect will be Selected at 20 and 50 feet to the Shoreline	Critical	(*)
Yaquina River Downstream of Hoy's Marine	SVOCs TOC PCBs Metals TBT	Confirm a Release of Hazardous Substances Attributable to Hoys Marine Site	Contaminated Surface Sediments	Four Samples in Two Transects at Two Samples per Transect will be Selected at 20 and 50 feet to the Shoreline	Critical	(*)
Off-Shore Wetland Intertidal Surface Sediment	SVOCs TOC Metals TBT	Confirm a release of hazardous substances to the river.	Contaminated Surface Sediments	Three Samples Along the Wetland Frontage will be Selected	Critical	(*)
Background Yaquina River Surface Sediment	SVOCs TOC PCBs Metals TBT	To Determine Background Concentrations	Background Surface Sediments	One Intertidal, Two Subtidal Surface Sediment Samples will Be Collected.	Critical	(*)
Off-Site Surface Soil	VOCs SVOCs PCBs Metals	To Determine Background Concentrations	Contaminated Surface Soil	Two Samples will be Selected	Critical	(*)

Key:

Critical = Required to achieve project objectives or limits on decision errors.
 NA = Not Applicable.
 Noncritical = For informational purposes only or needed to provide background information.
 PCBs = Polychlorinated biphenyls.
 (*) = TBT and TOC will be validated against QC criteria in the applicable analytical methods and the attached SOWs

Table 2-2

**SAMPLE ANALYSES SUMMARY
HOY'S MARINE SITE INSPECTION
NEWPORT, OREGON**

Location	Matrix ^a	Analytical Parameters and Method	Sample Preservation	Technical Holding Time ^b	Sample Container(s)
Former Waste Oil Tank Area	Surface Soil (2)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Former Solid Waste & Iron Shavings Dump Area	Surface Soil (2)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Bare Ground in Front of the Main Shop	Surface Soil (2)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Volatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	14 days from collection	Two 2-oz glass jars with Teflon-lined lids
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Paint Storage Shed	Surface Soil (4)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Volatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	14 days from collection	Two 2-oz glass jars with Teflon-lined lids
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid

Table 2-2

**SAMPLE ANALYSES SUMMARY
HOY'S MARINE SITE INSPECTION
NEWPORT, OREGON**

Location	Matrix ^a	Analytical Parameters and Method	Sample Preservation	Technical Holding Time ^b	Sample Container(s)
Between the Paint Storage Shed and Dry Dock	Surface Soil (1)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Volatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	14 days from collection	Two 2-oz glass jars with Teflon-lined lids
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Spent Grit Storage Shed	Surface Soil (2) Spent Grit (1)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Beneath Dry Dock (Intertidal)	Surface Sediments (4)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Volatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	14 days from collection	Two 2-oz glass jars with Teflon-lined lids
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Tributyltin Published Method by Krone et al	Cool to 4°C ± 2°C	Analyze within 14 days of collection	One 32-oz wide-mouth glass jar with Teflon-lined lid
		Total Organic Carbon USEPA SW-846 Method 9060	Cool to 4°C ± 2°C	Analyze within 28 days of collection	One 8-oz wide-mouth amber glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid

Table 2-2

**SAMPLE ANALYSES SUMMARY
HOY'S MARINE SITE INSPECTION
NEWPORT, OREGON**

Location	Matrix ^a	Analytical Parameters and Method	Sample Preservation	Technical Holding Time ^b	Sample Container(s)
Yaquina River at Hoys Marine (Subtidal)	Surface Sediments (12)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Tributyltin Published Method by Krone et al	Cool to 4°C ± 2°C	14 days from collection	One 32-oz wide-mouth glass jar with Teflon-lined lid
		Total Organic Carbon USEPA SW-846 Method 9060	Cool to 4°C ± 2°C	Analyze within 28 days of collection	One 8-oz wide-mouth amber glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Yaquina River Between Riverbend and Hoys Marine (Subtidal)	Surface Sediments (8)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Tributyltin Published Method by Krone et al	Cool to 4°C ± 2°C	14 days from collection	One 32-oz wide-mouth glass jar with Teflon-lined lid
		Total Organic Carbon USEPA SW-846 Method 9060	Cool to 4°C ± 2°C	Analyze within 28 days of collection	One 8-oz wide-mouth amber glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Yaquina River Downstream of Hoy's Marine (Subtidal)	Surface Sediments (4)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid

Table 2-2

**SAMPLE ANALYSES SUMMARY
HOY'S MARINE SITE INSPECTION
NEWPORT, OREGON**

Location	Matrix ^a	Analytical Parameters and Method	Sample Preservation	Technical Holding Time ^b	Sample Container(s)
Yaquina River Downstream of Hoy's Marine (Subtidal) (continued)	Surface Sediments (4) (continued)	Tributyltin Published Method by Krone et al	Cool to 4°C ± 2°C	14 days from collection	One 32-oz wide-mouth glass jar with Teflon-lined lid
		Total Organic Carbon USEPA SW-846 Method 9060	Cool to 4°C ± 2°C	Analyze within 28 days of collection	One 8-oz wide-mouth amber glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
Off-Shore Wetland (Intertidal)	Surface Sediments (3)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Tributyltin Published Method by Krone et al	Cool to 4°C ± 2°C	14 days from collection	One 32-oz wide-mouth glass jar with Teflon-lined lid
		Total Organic Carbon USEPA SW-846 Method 9060	Cool to 4°C ± 2°C	Analyze within 28 days of collection	One 8-oz wide-mouth amber glass jar with Teflon-lined lid
Background Surface Sediment (Upstream Yaquina River)	Surface Sediments (1 Intertidal and 2 Subtidal)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Tributyltin Published Method by Krone et al	Cool to 4°C ± 2°C	14 days from collection	One 32-oz wide-mouth glass jar with Teflon-lined lid
		Total Organic Carbon USEPA SW-846 Method 9060	Cool to 4°C ± 2°C	Analyze within 28 days of collection	One 8-oz wide-mouth amber glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Volatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	14 days from collection	Two 2-oz glass jars with Teflon-lined lids

Table 2-2

**SAMPLE ANALYSES SUMMARY
HOY'S MARINE SITE INSPECTION
NEWPORT, OREGON**

Location	Matrix ^a	Analytical Parameters and Method	Sample Preservation	Technical Holding Time ^b	Sample Container(s)
Background Surface Soil	Surface Soil (2)	Target Analyte List Metals (CLPAS) ILM04.0	Cool to 4°C ± 2°C	180 Days from collection (28 days for mercury)	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Volatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	14 days from collection	Two 2-oz glass jars with Teflon-lined lids
		Semivolatile Organic Compounds (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid
		Polychlorinated Biphenyls (CLPAS) OLM03.2	Cool to 4°C ± 2°C	Extract within 14 days of collection; analyze within 40 days of extraction	One 8-oz wide-mouth glass jar with Teflon-lined lid

^a The number of samples presented is an estimate; the actual number of samples to be collected will be determined in the field.

^b Technical holding times have been established only for water matrices. Water technical holding times were applied to sediment, soil, and product samples where applicable; in some cases, recommended sediment/soil holding times are listed.

Key:

C = Celsius.
 CLPAS = Contract Laboratory Program Analytical Service.
 HCl = Hydrochloric acid.
 L = Liter.
 ml = Milliliter.
 oz = Ounce.
 TBD = To be determined.

Table 2-3

**QA/QC ANALYTICAL SUMMARY AND FIXED LABORATORY ANALYTICAL METHODS
HOY'S MARINE SITE INSPECTION
NEWPORT, OREGON**

Laboratory	Matrix	Parameters/ Method	Method Description/ Detection Limits	Total Field Samples ^a / Containers	QA/QC Sample Summary Analyses / Containers				Total Field and QA/QC Analyses/ Containers ^e
					Trip Blanks ^b	Rinsate Blanks ^c	Organic MS/MSD ^d	Inorganic MS/Dup ^d	
EPA Region 10 or CLP Laboratory	Soil/ Sediment/ Grit	TAL Metals/ (CLPAS) ILM04.0	GFAA & ICP/ CRDL	50/ 50	0 / 0	1 / 1	NA	3 / 0	54 / 51
		Volatile Organic Compounds (CLPAS) OLM03.2	GC/MS & MD/ CRQL	14 / 28	1 / 2	0 / 0	1 / 0	NA	16 / 30
		Semivolatile Organic Compounds (CLPAS) OLM03.2	GCS & MD/ CRQL	50 / 50	0 / 0	1 / 1	3 / 0	NA	54 / 51
		Polychlorinated Biphenyls (CLPAS) OLM03.2	GCS & ECD/ CRQL	40 / 40	0 / 0	1 / 1	2 / 0	NA	43 / 41
Commercial Laboratory	Sediment	TBT/Published Method by Krone et al	GC/MS/ 2 mg/kg	34 / 34	0 / 0	1 / 1	NA	2 / 0	37 / 35
		TOC/USEPA SW-846 Method 9060	CA & FID/ 2 mg/kg	34 / 34	0 / 0	0 / 0	0 / 0	NA	34 / 34

^a Total number of field samples is estimated.

^b The total number of trip blanks could vary depending on the total number of sample shipments. This number is based on the estimated number of shipping containers.

^c The total number of rinsate samples could vary depending on the total number of samples collected. The sample numbers are based on one rinsate per 20 samples per nondedicated sampling device.

^d No extra volume is required for soil/sediment or product samples; for water samples, triple volume is required for organic analyses, and double volume is required for inorganic analyses. Sample numbers are based on 1 matrix spike/matrix spike duplicate (MS/MSD) per 20 samples per matrix.

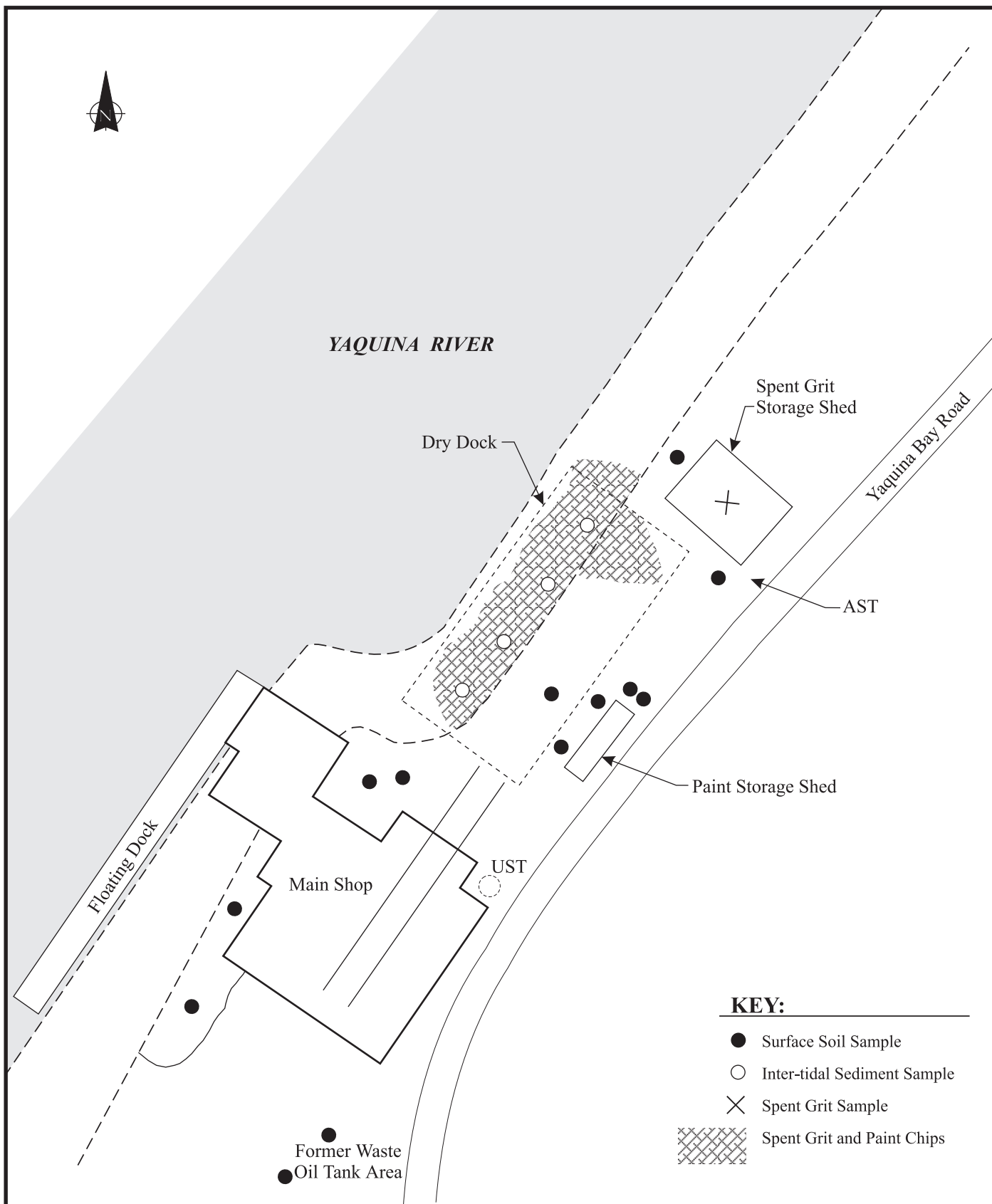
^e Total analyses and containers includes both field and QA/QC aliquots to be submitted for fixed laboratory analysis.

Key:

CLP = Contract Laboratory Program.
 CLPAS = Contract-Laboratory Program Analytical Service.
 CRDL = Contract-Required Detection Limit.
 CRQL = Contract Required Quantitation Limit.
 ECD = Electron Capture Detection.
 GFAA = Graphite Furnace Atomic Absorption.

GC/MS = Gas Chromatography/Mass Spectrometry.
 ICP = Inductively Coupled Plasma.
 MD = Mass Spectrometric Detection.
 MS/MSD = Matrix Spike/Matrix Spike Duplicate.
 NA = Not Applicable.
 QA = Quality Assurance.
 QC = Quality Control.
 TAL = Target Analyte List.

Table 2-4 SAMPLE CODING HOY'S MARINE SITE INSPECTION NEWPORT, OREGON		
Digits	Descriptions	Code Example
1,2	Source Code	BG (Background) IT (Intertidal) RS (Rinsate) ST (Sub-tidal) SS (On-Site Surface Soil) TB (Trip Blank) WL (Wetland))
3,4	Consecutive Sample Number	01 (First Sample of Source Code)
5,6	Matrix Code	GR (Spent Grit)
		SD (Sediment) SS (Soil) WT (QC Sample Water)



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HOY'S MARINE SITE
Newport, Oregon

Not to Scale

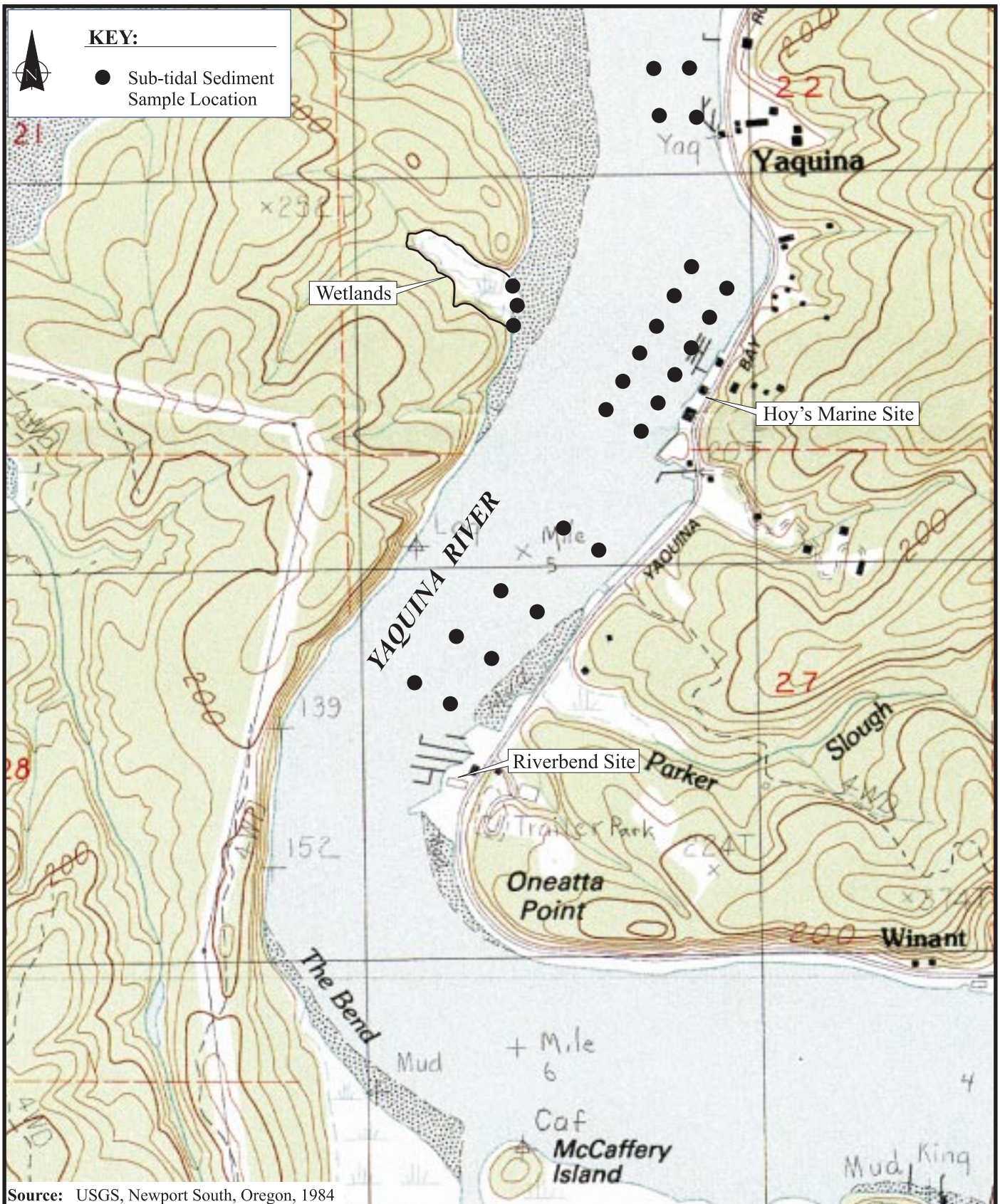
Figure 2-1
ON-SITE SAMPLE LOCATION MAP

Drawn:
AES

DATE:
4/19/99

JOB NO.
CG1101SIT0

Dwg.No.
CG1101 2-1



Source: USGS, Newport South, Oregon, 1984



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Seattle, Washington

HOY'S MARINE SITE Newport, Oregon

0 1000 2000
Approximate Scale in Feet

Figure 2-2 YAQUINA RIVER SAMPLE LOCATION MAP

Drawn: AES	DATE: 4/7/99	JOB NO. CG1101SIT0	Dwg.No. CG1101 2-2
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3. ASSESSMENT/OVERSIGHT

3.1 ASSESSMENTS AND RESPONSE ACTIONS

The EPA QA manager or designee may conduct an audit of the field activities for this project. The auditor will have the authority to issue a stop work order upon finding a significant condition that would adversely affect the quality and usability of the data. The EPA TM will have the responsibility for initiating and implementing response actions associated with findings identified during the site audit. Once the response actions have been implemented, the EPA QA manager or designee may perform a follow-up audit to verify and document that the response actions were implemented effectively. In-house audits performed by the START may be conducted in accordance with the E & E START *Quality Management Plan*. No audits are planned for the Hoy's Marine SI.

3.2 REPORTS TO MANAGEMENT

Debriefing of the EPA TM occurs by the START PM on a daily basis. Laboratory deliverables will be as specified in the CLP Organic and Inorganic Statements of Work (OLMO3.2 and ILMO4.0, respectively) for CLP and/or Manchester Environmental Laboratory data and as specified in the laboratory subcontract bid specification package for commercial laboratory analyses. Once the project is complete and the resulting data is obtained, the START PM will assist in preparation of a final project report. The report will include a summary of the activities performed during the project and the resulting data (along with any statements concerning data quality). The report will be approved by the EPA TM prior to forwarding to the individuals identified in the data distribution list located in the **Table of Contents** section of this SQAP.

4. DATA VALIDATION AND USABILITY

4.1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The data validation review of data packages will include an evaluation of: the information provided on the analytical data sheets and required support documentation for all sample analyses; the supporting sample collection documentation, including chain-of-custody; and field instrument calibration, results, and/or performance check documentation (if required by the method). The QA review will also examine adherence to the procedures as described in the cited SOPs and the requested analytical methods.

4.1.1 Data Reduction

Data reduction includes all processes that change the numerical value of the raw data. All fixed-laboratory data reduction will be performed in accordance with the appropriate methodology and will be presented as sample results.

4.1.2 Data Validation

Validation of CLP and/or Manchester Environmental Laboratory data generated under the Hoy's Marine SI will be performed by the EPA Region 10 QA office or its designee. Validation of data generated by a subcontracted laboratory will be performed by E & E and will examine adherence to the QC criteria outlined in the specific analytical methods. Data validation packages must be completed within normal turnaround of receipt of analytical data from the laboratory to meet the schedule prescribed by the EPA. If requested by the EPA, E & E will evaluate all data for precision, accuracy, and completeness before submission of the SI report to the EPA. Specific procedures for EPA Region 10 QA office (or its' designee) data validation to be used in this project are discussed in **Section 4** of the QMP and presented in the following EPA documents:

- *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (1994b); and
- *EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (1994c).

When not specifically covered by these guidelines, the analytical method QC requirements will also be utilized for validation. The QC parameters of interest for the EPA organic and inorganic methods that will be used on the Hoy's Marine SI samples are presented in these documents. In particular, data validation will include reporting of bias for all estimated data ("J" or "UJ" flags) using the following bias qualifiers:

- B = Detected concentration is below the method reporting limit/Contract-Required Detection Limit but is above the instrument detection limit;
- H = High bias;
- K = Unknown bias;
- L = Low bias; and
- Q = Detected concentration is below the method reporting limit/Contract-Required Quantitation Limit but is above the method detection limit.

If requested by the EPA, the validation deliverable generated by the EPA Region 10 QA office or its designee then will be reviewed by an E & E START chemist, focusing on the parameters listed in the EPA documents cited above.

The information to be included in the SI report will consist of a tabulation of analytical results and supporting QC information. Raw data will be available for inspection and maintained in the central job file.

4.1.3 Data Assessment Procedures

Following data validation and reporting, all project-generated and -compiled data and information will be reconciled with the objectives specified in [Section 1.3](#) to assess the overall success of SI activities. This data assessment, including points of achievement and departure from project-specific objectives, will be discussed in the QA section of the SI report.

4.2 VALIDATION AND VERIFICATION METHODS

The analytical QA Requirements and data validation requirements will be as specified in [Section 4.1.2](#) (EPA 1994b and 1994c).

The EPA TM will perform the final review and approval of the data. The EPA TM or QA officer will look at field duplicates, matrix spike/matrix spike duplicates, lab blanks, and lab duplicates to ensure that they are acceptable. The EPA TM will also compare the sample descriptions with the field sheets for consistency and will ensure that any anomalies in the data are appropriately documented.

4.2.1 Performance and System Audits

Performance and system audits will be conducted in accordance with the criteria described in **Section 3** of the QMP. No audits are scheduled for the Hoy's Marine SI field activities.

4.2.2 Preventive Maintenance

All field instruments and equipment used for analysis will be serviced and maintained only by qualified personnel. All instruments will be maintained by senior staff and/or electronics technicians. All repairs, adjustments, and calibrations will be documented in an appropriate logbook or data sheet that will be kept on file. The instrument maintenance logbooks will clearly document the date, the description of the problems, the corrective action taken, the result, and who performed the work.

All equipment used by E & E in the field is subject to standard preventive maintenance schedules established by corporate equipment protocols. When in use, equipment will be inspected at least twice daily, once before startup in the morning and again at the end of the work shift before overnight storage or return to the charging rack. Regular maintenance, such as cleaning of lenses, replacement of in-line filters, and removal of accumulated dust, is to be conducted according to manufacturer's recommendations and in the field as needed, whichever is appropriate. All performed preventive maintenance will be entered in the individual equipment's logbook and in the site field logbook.

In addition to preventive maintenance procedures, daily calibration checks will be performed at least once daily before use and recorded in the respective logbooks. Additional calibration checks will be performed as required. All logbooks will become part of either the permanent site file or the permanent equipment file.

4.2.3 Corrective Actions

The START corrective action program is addressed in **Section 3** of the QMP. Corrective actions will be conducted in accordance with these QMP specifications.

4.2.4 Quality Assurance Reports

Data quality assurance memoranda QA reports will be generated as part of the Hoy's Marine SI if the START is responsible for data validation. If the EPA Region 10 QA office or its designee performs the data validation, then additional reports regarding data usability will be generated by the START.

4.3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

Once the data results are compiled, the EPA TM will review the sample results to determine if they fall within the acceptance limits as defined in this SQAP. Completeness will also be evaluated to determine if the completeness goal for this project has been met. If data quality indicators do not meet the project's requirements as outlined in this SQAP, the data may be discarded and resampling and reanalysis may occur. The TM will attempt to determine the cause of the failure (if possible) and make the decision to discard the data and resample. If the failure is tied to the analysis, calibration and maintenance techniques will be reassessed as identified by the appropriate laboratory personnel. If the failure is associated with the sample collection and resampling is required, the collection techniques will be reevaluated as identified by the START PM.

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APPENDIX A
STANDARD OPERATING PROCEDURES



TITLE: SEDIMENT SAMPLING

CATEGORY: ENV 3.8

REVISED: March 1998

STANDARD OPERATING PROCEDURE

SEDIMENT SAMPLING

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ENV 3.8

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March 1998

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1. Introduction

This Standard Operating Procedure (SOP) describes the procedures for the collection of representative sediment samples. Analysis of sediment samples may determine whether concentrations of specific pollutants exceed established threshold action levels, and whether the concentrations of pollutants present a risk to public health, welfare, or the environment.

2. Scope

Included in this discussion are procedures for obtaining representative samples, quality assurance measures, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Sediment samples may be recovered using a variety of methods and equipment. These are dependent on 1) the depth of the water in which the samples will be collected; 2) the sediment's characteristics; 3) the volume of sediment required; and 4) the type of sample required (disturbed or undisturbed). Ultimately, the type of sampling device used should be consistent with the objective of the study.

Near-surface sediment samples may be collected using a scoop or spoon (if near shore or in shallow water), or sediment dredge or grab sampler (if in deeper water). To obtain other than surficial sediment samples, core samplers or split-spoon samplers are required.

All sampling devices should be cleaned using pesticide-grade acetone (assuming that acetone is not a target compound) or methanol, rinsed with distilled water, wrapped in aluminum foil, and custody sealed for identification. The sampling equipment should remain in this wrapping until needed. Each sampler should be used for one sample only. However, dedicated samplers may be impractical if there are a large number of sediment samples to be collected. In this case, samplers should be cleaned in the field using the decontamination procedures outlined in E & E's *Equipment Decontamination SOP*.



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4. Sample Preservation, Containers, Handling, and Storage

The chemical preservation of sediments is not generally recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time. Sediment samples should be handled according to standard techniques and project-specific requirements as detailed in project work/sampling plans and quality assurance project plans.

5. Potential Problems

Potential problems with sediment sampling include cross-contamination of samples and improper sample collection. Cross-contamination problems may be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then proper decontamination of sampling equipment is necessary. Improper sample collection can involve using inadequate or inappropriate sampling devices, contaminated equipment, disturbance of the matrix resulting in compaction of the sample, and inadequate homogenization of the sample where required, resulting in variable, nonrepresentative results.

6. Equipment

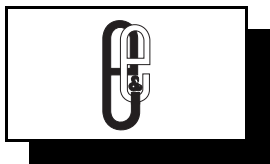
The following is a list of equipment and items typically used for sediment sampling:

- Sampling plan,
- Sample location map,
- Safety equipment, as specified in the health and safety plan,
- Compass,
- Survey equipment,
- Tape measure,
- Camera,



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- Four-ounce and eight-ounce glass jars with teflon liners,
- 40-ml glass vials with teflon-backed septum,
- Plastic bags for sample jars,
- Logbook,
- Labels,
- Waterproof ink pen,
- Chain-of-custody forms,
- Shipping cooler,
- Decontamination supplies and equipment, as described in the work plan,
- Canvas or plastic sheeting,
- Stainless-steel scoops,
- Stainless-steel spoons,
- Stainless-steel mixing bowls, or pans,
- Hand-driven split-spoon sampler,
- Shovel,
- Stainless-steel hand auger,
- Sediment dredge/grab sampler,
- Manual, gravity, or mechanical coring devices, and
- Teflon beaker attached to a telescoping pole.



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7. Reagents

Sediment sampling does not require the use of reagents except for decontamination of equipment. Refer to E & E's *Equipment Decontamination SOP* and the site-specific work plan for proper decontamination procedures and appropriate solvents.

8. Procedures

8.1 Office Preparation

- Prepare a sampling plan in accordance with contract requirements. Conduct a literature and information search and review available background information (e.g., topographic maps, soil survey maps, geological survey maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling methods to be employed, and the type and amounts of equipment and supplies required.
- E & E corporate policy requires that a health and safety plan be prepared prior to commencing any sampling activity. The plan must be approved and signed by the corporate health and safety officer or his/her designee (e.g., the regional safety coordinator [RSC]).
- Obtain necessary sampling and monitoring equipment (see Section 6), and ensure that everything is in working order.
- Contact delivery service to confirm ability to ship all equipment and samples. Determine whether shipping restrictions exist.
- Prepare schedules and coordinate with staff, clients, property owners, and regulatory agencies, if appropriate.

8.2 Field Preparation

- Identify local suppliers of sampling expendables and overnight delivery services (e.g., Federal Express).
- Decontaminate or preclean all equipment before sediment sampling, as described in E & E's *Equipment Decontamination SOP*, or as deemed necessary.



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- Calibrate all health & safety monitoring equipment daily.
- A general site survey should be performed prior to site entry, in accordance with the health and safety plan. A site safety meeting identifying physical and chemical hazards should be conducted prior to sampling activities.
- Identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All locations must be cleared of utilities by the property owner or utility companies prior to sediment sampling.

8.3 Sample Collection

Numerous techniques and sampling devices may be employed to collect representative sediment samples. A number of sampling-related factors can contribute to the loss of sample integrity, including washout of fine-grained sediments during retrieval; compaction due to sample wall friction; and sampling vessel- or person-induced disturbance of surficial layers. Choosing the most appropriate sediment sampler for a study will depend on the sediment's characteristics, the volume and efficiency required, and the objectives of the study.

Most samples will be grab samples, although occasionally, sediment taken from various locations may be combined into one composite sample to reduce the amount of analytical support required.

The following procedure is used to collect surface sediment samples from small, low-flowing streams or near the shore of a pond or lake:

1. The sampler should select the sampling location furthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments from migrating down to unsampled locations. This technique will also reduce the chances of cross-contaminating subsequent samples by sampling first in areas of suspected low contamination and working to the suspected higher concentration areas.
2. Using a precleaned, stainless-steel scoop, spoon, or other appropriate device, remove the required volume of sediment from the desired surface interval (e.g., 0-inch to 6-inch), place the sample in the appropriate precleaned glass jar, decant excess liquid as necessary, and secure the teflon-lined lid to the jar. If the sample is to be a composite sample, or if the sample is to be homogenized, the sediment is first placed in a stainless-steel mixing bowl and is homogenized prior to placement in the glass sample container. Samples for volatile organic analysis are not homogenized. Samples are handled in accordance with project-specific requirements.



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3. Carefully and clearly identify the jar with the appropriate sample label, ensuring that all the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, secure the seal in place with clear tape, and refrigerate the sample. The clear tape should also cover the jar's label.
4. Use the chain-of-custody form to document the types and number of sediment samples collected for shipment to a laboratory for analyses.
5. In the field logbook record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements.
6. Decontaminate sampling equipment in accordance with E & E's *Equipment Decontamination SOP*.

The following procedure is used to collect subsurface sediment samples from small, low-flowing streams or near the shore of a pond or lake:

1. The sampler should select the sampling location farthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments from migrating downstream to unsampled locations, and will also reduce the chances of cross-contaminating subsequent samples.
2. Using a precleaned split-spoon sampler or other hollow coring device, drive the sampler to the required depth with a smooth continuous motion. Remove the coring device by rotating and lifting it in a single smooth motion until the sampler is free from the sediment.
3. Before the sediment sample can be removed from the sampling device, the overlying water must be removed from the sampler by slowly pouring or siphoning it off near one side of the sampler. Care should be taken to ensure that the sediments are not disturbed, and that the fine-grained surficial sediment and organic matter are not lost while removing the overlying water.
4. Disassemble the split-spoon sampler by placing pipe wrenches on either end of the sampler. Remove both ends and open the split spoon with a precleaned stainless-steel spoon. Recover the sediment core from a core tube by pushing the sample out with a precleaned stainless-steel spoon.



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5. Collect the necessary sample by cutting the core with the handle of a precleaned stainless-steel spoon, placing the sample in the appropriate precleaned glass jar, and securing the teflon-lined lid to the jar. Samples are handled in accordance with project-specific requirements.
6. Carefully and clearly label the jar with the appropriate sample tag, ensuring that all of the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, and secure the seal in place with clear tape.
7. Use the chain-of-custody form to document the types and number of sediment samples collected and logged.
8. Record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements, in the field logbook.
9. Decontaminate sampling equipment as per E & E's *Equipment Decontamination SOP*.

The following procedure is used to collect surface samples from rivers or from deeper lakes and ponds:

1. The sampler should select the sampling location farthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments to migrate downstream to unsampled locations.
2. Using a precleaned sediment dredge or grab sampler, lower the sampler to the sediment layer with a polypropylene rope. Depending on the type of sampler used, the jaws of the sediment dredge will either automatically close, or will be triggered with a weighted messenger.
3. Recover the sampler and empty the sediment sample into a precleaned stainless-steel bowl. The water layer should be decanted slowly until only sediment remains in the bowl.
4. Using a precleaned stainless-steel spoon, remove the required volume of sediment. Place the sample in the appropriate precleaned glass jar, and secure the Teflon-lined lid to the jar.
5. Carefully and clearly identify the jar with the appropriate sample label, ensuring that all of the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, and secure



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the seal in place with clear tape. The clear tape should cover the sample label.

6. Use the chain-of-custody form to document the types and number of sediment samples collected for shipment to a laboratory for analyses.
7. Record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements, in the field logbook.
8. Decontaminate sampling equipment in accordance with E & E's *Equipment Decontamination SOP*.

The following procedure is used to collect subsurface samples from rivers or from deeper lakes and ponds:

1. The sampler should select the sampling location farthest downstream for the first sample and work upstream. This will reduce the potential for disturbed sediments to migrate downstream to unsampled locations.
2. Attach a precleaned gravity or mechanical coring device to the required length of polypropylene sample line and allow the corer to freefall through the water to the bottom.
3. Determine the depth of sediment penetration, and if acceptable, retrieve the corer with a smooth, continuous lifting motion.
4. Remove the overlying water from the corer by slowly pouring or siphoning it off near one side of the sampler. Remove the nosepiece from the corer, and slide the sample out of the corer into a stainless-steel bowl or tray.
5. Collect the necessary sample by cutting the core with the handle of a stainless-steel spoon, placing the sample in the appropriate precleaned glass jar, and securing the teflon-lined lid to the jar. Samples are handled in accordance with project-specific requirements.
6. Carefully and clearly label the jar with the appropriate sample tag, ensuring that all of the categories or parameters listed in Section 10.1.1 have been addressed. Place a custody seal on the jar and lid, and secure the seal in place with clear tape.



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7. Use the chain-of-custody form to document the types and number of sediment samples collected for shipment to a laboratory for analyses.
8. Record the time and date of sample collection, as well as a description of the sample and any associated air monitoring measurements, in the field logbook.
9. Decontaminate sampling equipment in accordance with E & E's *Equipment Decontamination SOP*.

8.4 Postoperations

1. Decontaminate all equipment according to E & E's *Equipment Decontamination SOP* prior to shipping the equipment back to the warehouse.
2. Organize field notes into the report format required by E & E's *Field Report Preparation SOP*. Logbooks should be maintained according to E & E's *Field Activities Log Book SOP*.
3. All samples should be shipped on the same day that they were collected to arrive at the laboratory not more than 24 hours after the samples were collected in accordance with E & E's *Sample Packaging SOP*.

9. Calculations

There are no specific calculations required for sediment sampling.

10. Quality Assurance

10.1 Sample Documentation

10.1.1 Sediment Sample Label

All sediment samples shall be documented in accordance with standard labeling techniques and project-specific requirements. The sediment sample label is completed to the fullest possible extent, prior to collecting the sample, and should contain the following minimum information:



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- Site name or identification;
- Sample location and identifier;
- Date sample was collected in a day, month, year format (e.g., 03 JUN 91 for June 3, 1991);
- Time of sample collection, using 24-hour clock in the hours: minutes format; and
- Analysis required.

10.1.2 Logbook

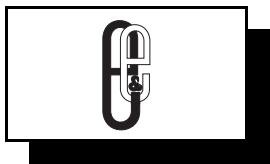
A bound field logbook will be maintained by field personnel to record daily activities in accordance with E & E's *Field Activities Logbooks* SOP and include sample collection, tracking, and shipping information. A separate entry will be made for each sample collected. These entries should include information from the sample label and a complete description of the location from which the sediment sample was collected.

10.1.3 Chain-of-Custody

Use the chain-of-custody form to document the types and number of sediment samples collected and logged.

10.2 Sampling Plan Design

- Many of the activities critical to ensuring that the collected samples are of high quality take place in the pre-collection planning and preparation stage. Careful planning and attention to detail at this stage will result in a more successful sampling effort, and will ensure collection of the highest quality samples possible. Since site and sampling conditions vary widely, and no universal sampling procedure can be recommended, a detailed sampling plan, consistent with the objectives of the study, must be developed prior to any sampling activities.
- Any of the sampling methods described here should allow a representative sediment sample to be obtained if the sampling plan is properly designed.
- Consideration must also be given to the collection of a sample representative of all horizons present in the sediment. Selection of the proper sampling device will facilitate this procedure.



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- A stringent quality assurance project plan (QAPP) should be outlined before any sampling operation is attempted. This should include, but not be limited to, the use of properly cleaned samplers and sample containers, chain-of-custody procedures, and collection of quality assurance samples such as field blanks, trip blanks, and duplicate samples.

11. Data Validation

The data generated will be reviewed according to quality assurance (QA) considerations identified in Section 10.

12. Health and Safety

Depending on site-specific contaminants, various protective programs must be implemented prior to sediment sampling. The site safety plan should be reviewed with specific emphasis placed on a protection program planned for direct contact tasks. Standard safe operating practices should be followed, including minimizing contact with potential contaminants in both vapor and solid matrix by using both respirators and disposable clothing.

Use appropriate safe work practices for the type of contaminant expected (or determined from previous sampling efforts):

Particulate or Metals Contaminants

- Avoid skin contact with and incidental ingestion of dust. Wash hands and other exposed skin areas routinely.
- Use protective gloves when collecting and handling the sediment samples.

Volatile Organic Contaminants

- Hexane acts as a carrier for a number of semivolatile organic compounds. The presence of hexane vapors in the air while decontaminating samplers indicates that the potential for exposure exists.
- If monitoring results indicate the presence of organic vapors, sampling activities must be conducted in Level C protection.



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- Acetone can penetrate some types of surgical gloves; use the appropriate gloves, such as Scorpio neoprene gloves, when handling acetone.

13. References

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SAMPLE PACKAGING

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ENV 3.16

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STANDARD OPERATING PROCEDURE

SAMPLE PACKAGING

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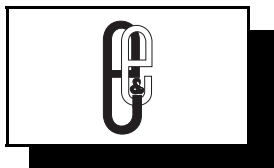
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1. Introduction

Liquid and solid environmental samples are routinely collected by E & E during field surveys, site investigations, and other site visits for laboratory analysis. Unless the samples have anesthetic, noxious, or other properties that could inhibit the ability of a flight crew member to perform his or her duty or are known to meet the established U.S. Department of Transportation criteria for hazardous material (i.e., explosive, corrosive, flammable, poisonous), they are not regulated as hazardous materials.

This Standard Operating Procedure (SOP) describes the packaging procedures to be used by E & E's staff to ensure the safe arrival of the samples at the laboratory for analyses. These procedures have been developed to reduce the risk of damage to the samples (i.e., breakage of the sample containers), promote the maintenance of sample temperature within the cooler, and prevent spillage of the sampled material should a container be broken.

In the event the sample material meets the established criteria of a DOT hazardous material, the reader is referred to E & E's Hazardous Materials/Dangerous Goods Shipping Guidance Manual (see H&S 5.5).

2. Scope

This SOP describes procedures for the packaging of environmental samples in:

- Coolers;
- Steel, aluminum and plastic drums; and
- 4GV fiberboard boxes.

The Hazardous Materials/Dangerous Goods Shipping Guidance Manual will complete the information needed for shipping samples by providing guidance on:

- Hazard determination for samples which meet the USDOT definition of a hazardous material;
- Shipping profiles for "standard" shipments;
- Shipping procedures for "non-standard" shipments;
- Marking of packages containing hazardous materials;



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- Labeling of packages containing hazardous materials; and
- Preparation of shipping papers for hazardous materials shipment.

3. Sample Packaging Procedures

3.1 General

It is E & E's intent to package samples so securely that there is no chance of leakage during shipment. This is to prevent the loss of samples and the expenditure of funds for emergency responses to spills and the efforts necessary to re-obtain the sample.

Over the years, E & E has developed several "standard" package configurations for the shipping of environmental samples. These standard package configurations are described below.

Liquid samples are particularly vulnerable. Because transporters (carriers) do not know the difference between a package leaking distilled water and a package leaking a hazardous chemical, they will react to a spill in an emergency fashion, potentially causing enormous expense to E & E for the cleanup of the sample material. Therefore, liquids are to be packed in multiple layers of plastic bags and absorbent/cushioning material to preclude any possibility of leaks from a package. This section defines the standard packaging configurations for environmental samples.

3.2 Liquid Environmental Sample Packaging Procedures

Liquid environmental samples should be collected and preserved as outlined in the Standard Operating Procedures (SOP) for Surface Water Sampling (ENV 3.12), and Groundwater Well Sampling (ENV 3.7). **Preserved water samples are not considered to meet the HM/DG definitions of Class 8 (Corrosive) and are therefore considered to be nonhazardous samples.** Liquid environmental samples may be shipped using an 80-quart cooler or an outer package consisting of either a steel or aluminum drum. Because the steel and aluminum drums provide little insulating capability, they should not be used for samples that require icing.

Packaging Liquid Environmental Samples Using the 80-Quart Cooler

- Label and seal all water sample bottles according to appropriate sampling SOPs;
- Secure the bottle caps using fiberglass tape; and
- Place each amber, poly, and volatile organic analysis (VOA) bottle in a sealable plastic bag. Mark the temperature blank VOA bag for identification.

If a foam block insert is used:

- Line the cooler with two plastic bags;



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- Place a foam insert (with holes cut to receive the sample bottles) inside the plastic bag;
- Place the bottles in the holes in the foam block;
- Fill void spaces with bagged ice to the top of the cooler;
- Fold over the plastic bags lining the cooler and secure shut with tape;
- Place Chain-of-Custody (C-O-C) form in a sealable bag and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the custody seals with clear tape.

If vermiculite is used:

- Place 1 inch of inert absorbent material (vermiculite) in the bottom of the cooler;
- Line the cooler with two plastic bags;
- Place each sample bottle inside the inner bag;
- Fill the void spaces around the bottles with vermiculite to about half the height of the large bottles;
- Fill the remainder of the void spaces with bagged ice to within 4 inches of the top of the cooler, making sure the VOAs are in direct contact with a bag of ice;
- Fold over the plastic bags lining the cooler and secure shut with tape;
- Fill the remaining space in the cooler with vermiculite to the top of the cooler;
- Place C-O-C form in a sealable bag and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the custody seals with clear tape.

Alternate Packaging Using 1A2/1B2 Drum

- Place 3 inches of inert absorbent material (vermiculite) in the bottom of the drum;
- Line the drum with two plastic bags;



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- Place each sample bottle inside the inner bag;
- Fill the void spaces around the bottles with vermiculite to the height of the larger bottles;
- Fold over the plastic bags lining the drum and secure shut with tape;
- Fill the remaining space in the drum with vermiculite to the top of the drum;
- Place C-O-C form in a sealable bag and tape it to the inside of the drum lid; and
- Secure the drum with closing ring and apply custody seals. Cover the custody seals with clear tape.

3.3 Soil/Sediment Environmental Sample Packaging Procedures

Soil/sediment environmental samples should be collected as outlined in the SOP for Soil Sampling (ENV 3.13), and SOP for Sediment Sampling (ENV 3.8). Soil/sediment environmental samples may be shipped using an 80-quart cooler, a 4GV fiberboard combination package, or an outer package consisting of either a steel or aluminum drum. Because the steel and aluminum drums provide little insulating capability, they should not be used for samples that require icing.

Packaging Soil/Sediment Environmental Samples

- Label and seal each sample container according to SOPs;
- Secure the bottle caps using fiberglass tape;
- Place each sample bottle inside a sealable plastic bag and place it in its original shipping box or in individual fiberboard boxes. Mark the temperature blank bag for identification; and
- Secure the original shipping box with strapping tape, place shipping box in a plastic bag, and secure the plastic bag with tape.

If an 80-quart cooler is used:

- Place bubble pack or similar material on the bottom and sides of an 80-quart cooler;
- Place the bagged shipping boxes in the cooler with a layer of bubble pack between each box;



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- Fill the void spaces with "blue ice" or ice in baggies to the top of the cooler;
- Place C-O-C form in a sealable baggie and tape it to the inside of the cooler lid; and
- Secure the cooler with strapping tape and custody seal. Cover the seals with clear tape.

If a 1A2/1B2 drum is used:

- Place 3 inches of inert absorbent material (vermiculite) in the bottom of the drum;
- Line the drum with two plastic garbage bags;
- Place the boxes inside the inner bag;
- Fill the space around the samples with vermiculite;
- Fold over the plastic bags lining the drum and secure shut with tape;
- Fill the remaining space around the bags with vermiculite to the top of the drum;
- Place C-O-C form in a sealable bag and tape it to the inside of the drum lid; and
- Secure the drum with the closing ring and apply custody seals. Cover the custody seals with clear tape.

Note: If a small number of samples are being shipped, it may be more practical to package them using the vermiculite or foam block configurations used for shipping liquid samples.

4. Shipping Procedures

Environmental samples are to be shipped as nonhazardous cargo. Unless the samples have anesthetic, noxious, or other properties that could inhibit the ability of a flight crew member to perform his or her duty or are known to meet the established U.S. Department of Transportation criteria for a hazardous material (i.e., explosive, corrosive, flammable, poisonous), they are not regulated as hazardous materials. When preparing the containers (i.e., cooler, drum, or box) for shipment, E & E staff **must** remove all labels from the outside container. Labels indicating that the contents may be hazardous are misleading and are not appropriate. Markings indicating ownership of the container, destination, and chain of custody labels are acceptable and can be attached as required.



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When completing the paperwork for shipment, the standard nonhazardous forms must be used. Do not use the hazardous materials/dangerous goods airbills, either in total or in part; these forms are coded and their use will invite unnecessary questions. This will only serve to confuse Airborne or Federal Express' terminal personnel and will cause much frustration and the delay of sample shipment.

Environmental sample packages can be shipped overnight by both Airborne and Federal Express. When choosing between the two, cost should be considered. It is normally much cheaper to ship Airborne. For work conducted and paid for by E & E, it is E & E's policy that you must first attempt to ship by Airborne before considering Federal Express. In addition, Airborne tends to have remote locations open later in the evenings than Federal Express, which may be helpful when trying to complete a full day's sampling effort and still make the flights on time. Although both companies offer pickup of samples at the site, it is advisable to call ahead and ensure that this service is offered beforehand. In almost all cases, both companies will deliver to the laboratory of your choice on Saturdays. When planning for sampling activities, check with the companies in advance to verify pick-up and delivery schedules.

When dropping off the sample packages at Airborne or Federal Express, do not indicate that these samples may be hazardous. This will only raise questions and delay the shipment. Although you may have an idea of the kind of contamination that is contained in the samples, you must remember that you are sending these samples to a laboratory for analyses to determine the amount and type of contamination, if any. Until that is determined, the presumption is that the samples are environmental in nature and not subject to the hazardous materials restrictions.



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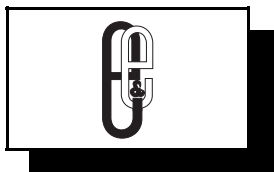
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1. Summary

This Standard Operating Procedure (SOP) establishes requirements for the entry of information into logbooks to ensure that E & E field activities are properly documented. The project manager (PM) and the field team leader (FTL) are responsible for ensuring that logbook entries provide sufficient information for the completion of an accurate and detailed description of field operations and meets the requirements of the contract or technical direction document (TDD).

This SOP describes logbook entry requirements for all types of projects, specifies the format that should be used, and provides examples. Some flexibility exists when implementing the SOP because different types of projects require different data collection efforts. This SOP does not address site safety logbook requirements or geotechnical logbook entries.

2. Purpose

Complete and accurate logbook entries are important for several reasons: to ensure that data collection associated with field activities is sufficient to support the successful completion of the project; to provide sufficient information so that someone not associated with the project can independently reconstruct the field activities at a later date; to maintain quality control (QC) throughout the project; to document changes to or deviations from the work plan; to fulfill administrative needs of the project; and to support potential legal proceedings associated with a specific project.

2.1 Adequate Field Information/Quality Control

QC procedures for data collection begin with the complete and systematic documentation of all persons, duties, observations, activities, and decisions that take place during field activities. It is especially important to fully document any deviations from the contract, project scope, work plans, sampling plans, site safety plans, quality assurance (QA) procedures, personnel, and responsibilities, as well as the reasons for the deviations.

Prior to entering the field, the project manager must indicate to the field team what pertinent information must be collected during field activity in order to meet the desired objectives of the data collection effort. The PM is responsible for reviewing the adequacy of the project logbooks both during and following completion of field activities, and is also responsible for meeting with the field team members to discuss any findings and to direct activities to correct any deficiencies, as appropriate. The PM also has the responsibility of ensuring that the logbooks become part of the project or TDD file.

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2.2 Work Plan Changes/Deviation

The logbook is the document that describes implementation of the work plan and other appropriate contract documents and provides the basis for the project reports. It must include detailed descriptions of any and all deviation from the work plan and the circumstances that necessitate such changes. These changes will be reviewed for compliance with data quality objectives and include:

- Changes in procedures agreed to in the project planning stages;
- Any conditions that prevent the completion of the field effort, or that result in additional fieldwork must be noted (i.e., weather delays, government actions, physical obstructions, personnel/equipment problems, etc.). Persons from whom permission was obtained to make such changes must be clearly documented.
- Any modifications requested by the client or client's representative that are contradictory to the contract or outside of the existing scope of work must be documented in detail because the cost of the project could be affected by such modifications.

2.3 Evidentiary Documentation

Field activity documentation can become evidence in civil and/or criminal judicial proceedings, as well as in administrative hearings. Field logbooks serve this purpose. Accordingly, such documentation is subject to judicial or administrative review. More importantly, it is subject to the review of an opposing counsel who will attempt to discredit its evidentiary value.

The National Enforcement Investigation Center (NEIC) and the United States Environmental Protection Agency (EPA) have prepared documents outlining their documentation needs for legal proceedings. These guidelines indicate the importance of accurate and clear documentation of information obtained during the inspections, investigations, and evaluations of uncontrolled hazardous waste sites. Consequently, attention to detail must be applied by E & E personnel to all field documentation efforts for all E & E projects. Project personnel must document where, when, how, and from whom any vital project information was obtained. This information is necessary to establish a proper foundation for admissible evidence.

3. Guidelines

Logbooks should contain a summary of any meeting or discussion held with a client or with any federal, state, or other regulatory agency that was on site during the field activities. The

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logbook should also describe any other personnel that appear on site, such as representatives of a potential responsible party (PRP).

The logbook can be used to support cost recovery activities. Data concerning site conditions must be recorded before the response activity or the passage of time eliminates or alters those conditions. Logbooks are also used to identify, locate, label, and track samples and their final disposition. In addition, data recorded in the logbook will assist in the interpretation of the analytical results.

Logbooks are subject to internal and external audits. Therefore, the recorded information should be consistent with and capable of substantiating other site documentation such as time cards, expense reports, chain-of-custody forms, shipping papers, and invoices from suppliers and subcontractors, etc. Logbooks also act as an important means of reconstructing events should other field documents such as data collection forms become lost or destroyed. Therefore, all mission-essential information should be duplicated in the logbook.

3.1 General Instructions

The following general guidelines must be used for all logbooks:

- At a minimum, one separate field activity logbook must be maintained for each project or TDD.
- All logbooks must be bound and contain consecutively numbered pages.
- No pages may be removed for any reason, even if they are partially mutilated or illegible.
- All field activities must be recorded in the site logbook (e.g., meetings, sampling, surveys, etc.).
- All information must be **printed legibly** in the logbook using waterproof ink, preferably black. If weather conditions do not permit this (i.e., if it is too cold or too wet to write with ink), another medium, such as pencil, may be used. The reason that waterproof ink was not used should be specifically noted in the logbook.
- The language used in the logbook should be objective, factual, and free of personal feelings or terminology that might prove inappropriate.
- Entries should be made in chronological order. Contemporaneous entries are always preferred because recollections fade or change over time. Observations that cannot be recorded during field activities should be recorded as soon after as possible. If logbook entries



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are not made during field activities, the time of the activity/observation and the time that it is recorded should be noted.

- The first entry for each day will be made on a new, previously blank page.
- Each page should be dated and each entry should include the time that the activity occurred based on the 24-hour clock (e.g., 0900 for 9 a.m., 2100 for 9 p.m.).
- At the completion of the field activity, the logbook must be returned to the permanent project or TDD file.

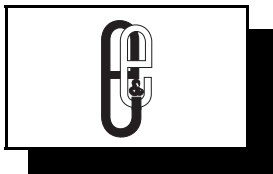
3.2 Format

The information presented below is not meant to be all-inclusive. Each project manager is responsible for determining the specific information requirements associated with a field activity logbook. If someone other than the Project Manager is keeping the logbook, the Project Manager is responsible to convey to that individual, prior to the start of fieldwork, specific instructions on what type of information is required to be entered into the logbook. Information requirements will vary according to the nature and scope of the project. (Refer to Appendix A for an example of a completed logbook.)

Title Page

The logbook title page should contain the following items:

- Site name,
- Location,
- TDD No. or Job No.,
- PAN (an EPA site/task identification number), if applicable,
- SSID No. (Site ID number-assigned under CERCLA), if applicable,
- Start/Finish date, and
- Book ___ of ___.



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First Page

The following items should appear on the first page of the logbook prior to daily field activity entries:

- TDD No. or Job No.,
- Date,
- Summary of proposed work (Reference work plan and contract documents, as appropriate),
- Weather conditions,
- Team members and duties, and
- Time work began and time of arrival (24-hour clock).

Successive Pages

In addition to specific activity entries and observations, the following items should appear on every logbook page:

- Date,
- TDD or Job No., and
- Signature (bottom of each page). If more than one person makes entries into the logbook, each person should sign next to his or her entry.

Last Page

In addition to specific activity entries and observations and the items that should appear on each successive page, the last page of the logbook should contain a brief paragraph that summarizes the work that was completed in the field. This summary can become especially important later on if more or less work was accomplished during the duration of the field activity.

3.3 Corrections

If corrections are necessary, they must be made by drawing a single line through the original entry in such a manner that it can still be read. *Do not erase or render an incorrect notation illegible.* The corrected entry should be written beside the incorrect entry, and the



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correction must be initialed and dated. Most corrected errors will require a footnote explaining the correction.

4. Documentation

Although the requirements and content of the field logbook will vary according to the site and the tasks to be performed, the following information should be included in every logbook:

4.1 Prior to Fieldwork

Summary of Proposed Work

The first paragraph of **each** daily entry should summarize the work to be performed on that day. For example:

"Collect soil and groundwater samples from previously installed wells and ship samples to Analytical Services Center (ASC). Discuss removal with site owner."

The first paragraph becomes especially important later when discussing work plan deviations or explaining why more or less work was accomplished for that day.

Personnel

Each person to be involved in activities for the day, his/her respective role (sampler, health and safety, etc.), and the agency he/she represents should be noted in the logbook.

On-Site Weather Conditions

Weather conditions may have an impact on the work to be performed or the amount of time required to perform the proposed work; therefore, all weather on-site weather conditions should be noted, including temperatures, wind speed and direction, precipitation, etc., and updated as necessary. Similarly, any events that are impacted by weather conditions should be noted in the logbook.

Site Safety Meeting

Although minutes should be recorded for all site safety meetings under separate cover, the logbook should briefly summarize the site safety meeting and any specific site conditions and resultant site safety concerns.



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4.2 Site Sketch

A site sketch should be prepared on the first day of field activities to indicate prominent site and environmental features. The sketch should be made either to scale or by noting the approximate distances between site feature. Area-specific sketches should be prepared as work is undertaken in such areas, and updated sketches should be drawn as work progresses.

Site Features

Examples of features to be noted on the site sketch include the following:

- Structures such as buildings or building debris;
- Drainage ditches or pathways, swales, and intermittent streams (include direction of overland runoff flow and direction of stream flow);
- Access roads, site boundaries, and utility locations;
- Decontamination and staging areas;
- Adjacent property data: the type of property that borders the site, information pertaining to ownership, and available addressees; and
- North arrow.

Changes in Site Conditions

Any deviation from previous site sketches or drawings presented in the work plan, and any changes that have occurred since the last site visit must be noted. Differences to be noted include the following:

- Demolished buildings;
- Changes to access routes;
- Damage to wells or equipment, or changes to the amount of such equipment believed to be on site,
- Changes resulting from vandalism;
- Destruction of reference points;

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- Changes resulting from environmental events or natural disasters; and
- Locations of excavations, waste piles, investigation-derived waste (IDW), drum staging areas, etc.

In short, *any* site condition that varies from the conditions described in the work plan should be noted.

4.3 Monitoring Equipment and Activities

Any monitoring equipment used during field activities should be documented in the logbook. Information to be noted includes:

- The type of equipment with model and serial numbers. (HNu, OVA, etc.);
- The frequency at which monitoring is performed;
- Calibration results and the frequency at which the equipment is calibrated or tested;
- Background readings;
- Any elevated or unusual readings; and
- Any equipment malfunctions.

It is particularly important to note elevated or unusual equipment readings because they could have an impact on personal protection levels or the activities to be performed on site. If a change in the proposed work or protection levels occurs, it should be clearly noted in the logbook.

4.4 Sample Collection Activities

Because it represents the first step in an accurate chain-of-custody procedure, field sampling documentation must be complete. The following items should be documented in the logbook:

Sample Collection Procedures

The following items pertaining to sample collection procedures should be included in the logbook:

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- Any pre-sampling activities (i.e., well purging and the number of volumes purged before sample collection);
- Results of the pre-sampling activities (i.e., pH/conductivity/temperature readings for well water, results of hazard categorization testing, etc.);
- Any environmental conditions that make sample collection difficult or impossible (i.e., dry or flooded drainage paths, inclement weather conditions, etc.); and
- Any deviation from the work plan (i.e., additional samples and the reason for their collection, alternate sample locations, etc.).

Sample Information

The following information regarding sample data should be recorded in the logbook:

- Sample number and station location including relationship to permanent reference point(s);
- Name(s) of sampler(s);
- Sample description and any field screening results;
- Sample matrix and number of aliquots if a composite sample;
- Preservatives used, recipient laboratory, and requested analyses;
- QA/QC samples; and
- Shipping paper (airbill) numbers, chain-of-custody form numbers, and jar lot numbers.

Investigation-Derived Waste/Sample Shipment

Details pertaining to sampling equipment, decontamination, and IDW should be clearly delineated in the work plan. However, the following information should be included in the logbook:

- The type of IDW generated and the number of containers generated (each drum should be numbered and its contents noted);
- All information relevant to the characterization of the IDW;

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- Any directions received from the client/workplan/contract relative to the management of the IDW;
- The disposition of IDW (left on site or removed from site);
- The number of sample containers shipped to the ASC or laboratory and the courier used (i.e., Federal Express, Airborne Express, etc.);
- Airbill or shipment tracking numbers; and
- The type of paperwork that accompanied the waste/sample shipment (e.g., manifests, etc.).

4.5 Photodocumentation

Photographs should be taken during all relevant field activities to confirm the presence or absence of contaminants encountered during fieldwork. Specific items to be documented include:

- Sample locations and collection activities;
- Site areas that have been disturbed or impacted, and any evidence of such impacts (i.e., stressed vegetation, seepage, discolored water, or debris);
- Hazardous materials requiring disposal, including materials that may not appear in the work plan;
- Any evidence that attests to the presence or absence of contamination; and
- Any features that do not appear in the work plan or differ from those described in the work plan.

Documentation of any photographs taken during the course of the project must be provided in the logbook with a detailed description of what is shown in the photograph and the reason for taking it. This documentation should include:

- Make, model, and serial numbers of the camera and lens,
- Film type and number of exposures,
- Roll and frame number of the photograph,



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- Direction or view angle of the photograph, and
- Name of the photographer.

4.6 Data Collection Forms

Certain phases of fieldwork may require the use of project-specific data collection forms, such as task data sheets or hazard categorization data sheets. Due to the specific nature of these forms, the information that should be included in the logbook cannot be fully discussed in this SOP. However, the following data should be included in the logbook:

- Results of any field tests or hazard categorization tests (i.e., ignitability, corrosivity, reactivity, etc.);
- The source from which any field sample was collected and its condition (i.e., drum, tank, lagoon, etc.).
- Other conclusions as a result of the data collected on data collection forms.

In many cases, rubber stamps that contain routine data collection forms can be manufactured ahead of time. These forms can be stamped into the logbook on an as-needed basis.



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Appendix A

Sample Logbook

RI 6130

WEDNESDAY JANUARY 26, 1994

PROPOSED WORK FOR DAY: COLLECT GROUNDWATER

SAMPLES FROM WELLS AND PIEZOMETERS AT SITE 1 AND SITE 3. SHIP SAMPLES TO THE ASC. CONTAINERIZE PURGE WATER. MEET WITH FRED CANSLER AND DISCUSS REMOVAL OF CANOPIES AT SITES 1 AND 3 AND FILLING OF EXCAVATIONS.

WEATHER ON SITE: CLOUDY AND WARM WITH A HIGH TEMPERATURE OF 50° F. RAIN SHOWERS WITH WINDS FROM THE SW AT 5-15 MPH.

EIE PERSONNEL ON SITE: G. JONES, J. MAYS, S. MC CONE

LOG

1330 ARRIVED ON SITE. THE GROUNDWATER SAMPLING CREW WAS PREPARING TO PURGE THE WELLS AND PIEZOMETERS IN THE FIELD ACROSS THE ROAD FROM SITE 1. PURGING OF WELLS BEING COMPLETED WITH HAND BAILERS SINCE PUMP IS IMPERATIVE.

1340 ARRIVED AT SITE 3. MW3-1 AND MW3-3 UNLOCKED AND OPEN. SECURED BOTH WELLS.
38 *Sumline 1/26/94*

RI 6130

1/26/94

1330 FRED CANSLER ARRIVED ON SITE. DISCUSSED

REMOVAL OF CANOPIES AND CLOSURE OF EXCAVATIONS

AT SITES 1 AND 3. FRED CANSLER STATED THAT

HE HAS A SOURCE FOR THE ROCK AND FOR THE TOP SOIL FOR THE EXCAVATIONS.

1405 ARRIVED AT THE SITE WHERE FRED CANSLER

PROPOSES TO REMOVE THE FILL FOR THE EXCAVATIONS.

A HILL ON THE WEST SIDE ⁴ OF THE WOODEN NICKLE IS IN THE PROCESS OF BEING REMOVED.

THE ROCK CONSISTS OF WEATHERED SHALE SIMILAR TO THE ROCK REMOVED FROM THE EXCAVATIONS.

FRED CANSLER PROPOSES TO USE THE ROCK TO FILL THE EXCAVATIONS TO WITHIN ONE FOOT OF GRADE.

1415 ARRIVED AT THE SITE WHERE FRED CANSLER

PROPOSES TO REMOVE TOP SOIL FOR THE EXCAVATIONS.

TOP SOIL REMOVED FROM THE YELLOW FREIGHT

LOT IS IN PILES ON THE NORTH SIDE OF THE LOT.

1430 RETURNED TO SITE 3. FRED CANSLER WILL

ARRANGE TO REMOVE THE CANOPY OVER

THE EXCAVATION AT SITE 3 ON THURSDAY

MORNING AND WILL ARRANGE TO BRING

THE ROCK IN ON THURSDAY AFTERNOON.

TWO TRUCKS WILL BE USED TO HAUL THE

FILL. THE SUPPORTS HOLDING THE CANOPY

39 *Sumline 1/26/94*

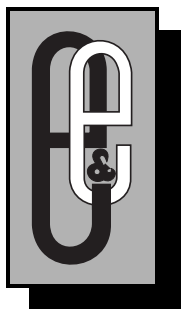
1/26/94 RI 6130
 1430 (cont) WILL BE CUT AND THE CANOPY DRAGGED AWAY FROM THE EXCAVATION.
 1445 CONTACTED JOY INMAN FROM ENVIRONICS. TANKERS WILL BE ON SITE ON THURSDAY TO PUMP OUT THE EXCAVATION AT SITE 3 AND ON FRIDAY TO REMOVE WATER AT SITE 1. A FRAC TANK WILL BE DELIVERED TO SITE 1 ON THURSDAY.
 1515 SAMPLING CREW COMPLETED PACKING SAMPLES COLLECTED AT SITE 1. ALL WELLS AND PIEZOMETERS AT SITE 1 HAVE BEEN SAMPLED.
 1530 SAMPLING CREW COMPLETED PACKING SAMPLES AND SECURING DRUMS OF PURGE WATER.
 1535 SAMPLING CREW DEPARTED SITE TO DELIVER SAMPLES TO FEDERAL EXPRESS.
 1600 CONTACTED TIM GRADY FROM E+E. DISCUSSED CONVERSATION WITH FRED CANSLER AND STATUS OF WELL/PIEZOMETER SAMPLING.
 1615 SECURED FOR DAY.
 WORK COMPLETED: COLLECTED GROUNDWATER SAMPLES FROM SITE 1 WELLS AND PIEZOMETERS. DISCUSSED REMOVAL OF CANOPIES AND FILLING OF EXCAVATIONS WITH FRED CANSLER. SHIPPED SAMPLES TO ASC.

John R. Jones
 1/26/94
 40

THURSDAY JANUARY 27, 1994 RI 6130
 PROPOSED WORK FOR DAY: COMPLETE COLLECTION OF GROUNDWATER SAMPLES AT SITE 3 AND SHIP THE SAMPLES TO THE ASC. REMOVE THE CANOPIES COVERING THE EXCAVATIONS AT SITES 1 AND 3. PUMP THE WATER OUT OF THE EXCAVATIONS AT SITES 1 AND 3 AND SHIP THE WATER OFF SITE TO OSCO. BACKFILL THE EXCAVATION AT SITE 3. REMOVE THE DRUMS FROM THE ROLL OFF BOX AND TRANSFER THE DRUMS TO THE WAREHOUSE.
 WEATHER ON SITE: CLOUDY AND COOL WITH A HIGH TEMPERATURE OF 45°F. WINDS VARIABLE 10-20 MPH.
 E+E PERSONNEL ON SITE: G. JONES, J. MAYS, S. MCONE
 LOG
 0700 SCOTT MCONE ARRIVED AT SITE 3.
 0710 ENVIRONICS PERSONNEL ARRIVED AT SITE 3.
 0715 HELD SITE SAFETY MEETING. DISCUSSED PHYSICAL AND CHEMICAL HAZARDS ASSOCIATED WITH SITE AND PROPOSED WORK FOR THE DAY.
 0725 E+E SAMPLING TEAM ARRIVED ON SITE.
 41 *John R. Jones* 1/27/94

1/27/94 RI 6130
 0730 E+E SAMPLING CREW COMMENCED COLLECTING
 SAMPLES AND PURGING MW 3-1 AND MW 3-2.
 0800 FRED CANSLER ARRIVED ON SITE WITH
 PERSONNEL TO REMOVE THE CANOPY OVER
 THE EXCAVATION AT SITE 3. THE SUPPORTS
 WERE CUT AND THE CANOPY WAS DRAGGED
 AWAY FROM THE EXCAVATION WITH TWO
 TRACTORS.
 0845 THE CANOPY REMOVAL AT SITE 3 COMPLETED
 AND THE CREW DEPARTED FOR SITE 1.
 0850 COMMENCED PUMPING WATER FROM THE
 EXCAVATION INTO BRYSON TRAILER # 618CS.
 0915 THE E+E SAMPLING TEAM COMPLETED COLLECTING
 THE GROUNDWATER SAMPLES FROM MW 3-1,
 MW 3-2, MW 3-3, AND MW 3-4. COMMENCED
 PACKING SAMPLES.
 0935 COMPLETED FILLING BRYSON TRAILER # 618CS
 WITH 5,000 GALLONS OF WATER AND PREPARED
 MANIFEST # 00941 FOR LOAD. COMMENCED
 LOADING BRYSON TRAILER # 429.
 1000 E+E SAMPLING TEAM DEPARTED THE SITE
 TO DELIVER SAMPLES TO FEDERAL EXPRESS.
 1030 ARRIVED AT SITE 1. THE CANSLER CREW
 IS IN THE PROCESS OF REMOVING THE
 CANOPY OVER THE EXCAVATION. CANOPY
 IS NOT MOVING AS A UNIT.
 42 *John C. 1/27/94*

RI 6130 1/27/94
 1045 RETURNED TO SITE 3. ALL WATER IN THE
 EXCAVATION HAS BEEN REMOVED EXCEPT
 FOR THE ICE. BRYSON TRAILER # 429
 LOADED WITH 5,200 GALLONS OF WATER. PREPARED
 MANIFEST # 00942 FOR LOAD. BOTH TRAILERS
 DEPARTED THE SITE.
 1100 ENVIRONICS PERSONNEL OPENED THE DRUMS
 OF DRILLING FLUIDS, DEVELOPMENT WATER
 AND PURGE WATER AND FOUND THE DRUMS
 FULL OF ICE. ENVIRONICS WILL CONTACT
 GARY SHOCKLEY AND RECOMMEND THAT
 THE DRUMS OR LIQUIDS BE TRANSPORTED
 TO OSED FOR TREATMENT SINCE THEY
 CAN NOT BE BULKED.
 1200 CANSLER CREW COMMENCED LOADING TRUCKS
 WITH STONE FROM THE SITE WEST OF
 THE WOODEN NICKEL.
 1230 ARRIVED AT THE SITE WHERE THE STONE
 WAS BEING LOADED. THE FILL MATERIAL
 IS ALL UNDISTURBED WEATHERED BEDROCK.
 1245 ARRIVED AT SITE 3. TWO LOADS OF
 ROCK FILL HAVE BEEN DUMPED IN THE
 EXCAVATION; AN ESTIMATED FOUR MORE
 LOADS OF STONE WILL BE NEEDED TO
 FILL THE EXCAVATION.
 1300 ARRIVED AT SITE 1. BRYSON TRAILER # 617
 43 *John C. 1/27/94*



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STANDARD OPERATING PROCEDURE

SOIL SAMPLING

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1. Introduction

This document describes the procedures for the collection of representative soil samples. Representative sampling ensures the accurate characterization of site conditions. Analysis of soil samples may determine pollutant concentrations and the accompanying risks to public health, welfare, or the environment.

2. Scope

Included in this discussion are procedures for obtaining representative samples, quality assurance/quality control (QA/QC) measures, proper documentation of sampling activities, and recommendations for personnel safety.

3. Method Summary

Soil samples may be recovered using a variety of methods and equipment. These are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type.

Samples of near-surface soils may be easily obtained using a spade, stainless-steel spoon, trowel, or scoop. Sampling at greater depths may be performed using a hand auger, a power auger, or, if a test pit is required, a backhoe.

All sampling devices should be cleaned using pesticide-grade acetone (assuming that acetone is not a target compound) or methanol, then wrapped in clean aluminum foil, and custody sealed for identification. The sampling equipment should remain in this wrapping until it is needed. Each sampler should be used for one sample only. However, dedicated tools may be impractical if there are a large number of soil samples required. In this case, samplers should be cleaned in the field using standard decontamination procedures as outlined in E & E's SOP for Equipment Decontamination (see ENV 3.15).

4. Sample Preservation, Containers, Handling, and Storage

The chemical preservation of solids is not generally recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time.

Soil samples should be handled according to the procedures outlined in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).

5. Potential Problems

Potential problems with soil sampling include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and bottles. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection is generally the result of the use of

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contaminated equipment; the disturbance of the matrix, resulting in compaction of the sample; and inadequate homogenization of the sample where required, resulting in variable, nonrepresentative results. Specific advantages and disadvantages of soil sampling equipment are presented in Table 5-1.

Table 5-1		
SOIL SAMPLING EQUIPMENT		
Equipment	Applicability	Advantages and Disadvantages
Trier	Soft surface soil	Inexpensive; easy to use and decontaminate; difficult to use in stony, dry, or sandy soil.
Scoop, trowel, spoon, or spatula	Soft surface soil	Inexpensive; easy to use and decontaminate; trowels with painted surfaces should be avoided.
Tulip bulb planter	Soft soil, 0 to 6 inches	Easy to use and decontaminate; uniform diameter and sample volume; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; not useful for hard soils.
Spade or shovel	Medium soil, 0 to 12 inches	Easy to use and decontaminate; inexpensive; can result in sample mixing and loss of VOCs.
Vehimeyer soil outfit	Soil, 0 to 10 feet	Difficult to drive into dense or hard material; can be difficult to pull from ground.
Soil coring device and auger	Soft soil, 0 to 24 inches	Relatively easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); limited depth capability; can be difficult to decontaminate.
Thin-walled tube sampler	Soft soil, 0 to 10 feet	Easy to use; preserves soil core (suitable for VOA and undisturbed sample collection); may be used to help maintain integrity of VOA samples; easy to decontaminate; can be difficult to remove cores from sampler.
Split-spoon sampler	Soil, 0 inches to bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); acetate sleeve may be used to help maintain integrity of VOA samples; useful for hard soils; often used in conjunction with drill rig for obtaining deep cores.
Shelby tube sampler	Soft soil, 0 inches to bedrock	Excellent depth range; preserves soil core (suitable for VOA and undisturbed sample collection); tube may be used to ship sample to lab undisturbed; may be used in conjunction with drill rig for obtaining deep cores and for permeability testing; not durable in rocky soils.
Laskey sampler	Soil, 0 inches to bedrock	Excellent depth range; preserves soil cores; used in conjunction with drill rig for obtaining deep core; can be difficult to decontaminate.
Bucket auger	Soft soil, 3 inches to 10 feet	Easy to use; good depth range; uniform diameter and sample volume; acetate sleeve may be used to help maintain integrity of VOA samples; may disrupt and mix soil horizons greater than 6 inches in thickness.
Hand-operated power auger	Soil, 6 inches to 15 feet	Good depth range; generally used in conjunction with bucket auger for sample collection; destroys soil core (unsuitable for VOA and undisturbed sample collection); requires two or more equipment operators; can be difficult to decontaminate; requires gasoline-powered engine (potential for cross-contamination).
Continuous-flight auger	Soil, 0 inches to bedrock	Excellent depth range; easy to decontaminate; can be used on all soil samples; results in soil mixing and loss of VOCs.

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Table 5-1**SOIL SAMPLING EQUIPMENT**

Equipment	Applicability	Advantages and Disadvantages
Dutch auger	Designed specifically for wet, fibrous, or rooted soils (e.g., marshes)	
Eijkelcamp stoney soil auger	Stoney soils and asphalt	
Backhoe	Soil, 0 inches to 10 feet	Good depth range; provides visual indications as to depth of contaminants; allows for recovery of samples at specific depths; can result in loss of VOCs and soil mixing; shoring required at depth.

Note: Samplers may not be suitable for soils with coarse fragments.
Augers are suitable for soils with limited coarse fragments; only the stoney auger will work well in very gravelly soil.

6. Soil Sampling Equipment

Soil Sampling Equipment List

- Stainless-steel spoon
- Trier
- Scoop
- Trowel
- Spatula
- Stainless-steel tulip bulb planter
- Spade or shovel
- Vehimeyer soil sampler outfit
 - tubes
 - points
 - drive head
 - drop hammer
 - fuller jack and grip
- Soil-coring device
- Thin-walled tube sampler
- Split-spoon sampler
- Shelby tube sampler
- Laskey sampler
- Bucket auger
- Hand-operated power auger
- Continuous-flight auger
- Dutch auger
- Eijkelcamp stoney soil auger
- Backhoe
- Hand auger with replaceable sleeves



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Sampling Support Equipment and Documentation List

- Sampling plan
- Sample location map
- Safety equipment, as specified in the Health and Safety Plan
- Decontamination supplies and equipment, as described in the Work Plan
- Compass
- Tape measure
- Survey stakes or flags
- Camera
- Stainless-steel buckets or bowls
- Sample containers, precleaned (i.e., I-Chem)
- Logbook
- Chain-of-custody forms
- Plastic sheet
- Soil gas probes
- Infiltrometer
- Pounding sleeve
- Extension rods
- T-handle

Labeling, Packaging, and Shipping Supplies

- Coolers
- Labels for sample containers and coolers (e.g., "fragile")
- Ice
- Plastic bags for sample containers and ice
- ESC paint cans and clamps for polychlorinated biphenyl (PCB) sampling
- Vermiculite
- Duct and strapping tape
- Federal Express airbills and pouches

6.1 Geophysical Equipment

Geophysical techniques can be integrated with field analytical and soil sampling equipment to help define areas of subsurface contamination. For a description of the geophysical techniques and associated applications, refer to E & E's SOP for Surface Geophysical Techniques (see GEO 4.2).

7. Reagents

This procedures does not require the use of reagents except for decontamination of equipment, as required. Refer E & E's SOP for Equipment Decontamination (see ENV 3.15) and the Site-Specific Work Plan (SSWP) for proper decontamination procedures and appropriate solvents.



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8. Procedures

8.1 Office Preparation

1. The preparation of a Health and Safety Plan is required prior to any sampling. The plan must be approved and signed by the Corporate Health and Safety Officer or his/her designee (i.e., the Regional Safety Coordinator [RSC]).
2. Prepare a sampling plan to meet the data quality objectives (DQO) of the project in accordance with contract requirement. Review available background information (i.e., topographic maps, soil survey maps, geologic maps, other site reports, etc.) to determine the extent of the sampling effort, the sampling method to be employed, and the type and amounts of equipment and supplies required.
3. Obtain necessary sampling and monitoring equipment (see Section 6), decontaminate or preclean the equipment, and ensure that it is in working order.
4. Contact delivery service to confirm ability to ship all equipment and samples. Determine if shipping restrictions exist.
5. Prepare schedules and coordinate with staff, clients, and regulatory agencies, if appropriate.

8.2 Field Preparation

1. Identify local suppliers of sampling expendables (e.g., ice, plastic bags) and overnight delivery services (e.g., Federal Express).
2. Decontaminate or preclean all equipment before soil sampling, as described in E & E's SOP for Equipment Decontamination (see ENV 3.15), or as deemed necessary.
3. A general site survey should be performed prior to site entry in accordance with the Health and Safety Plan followed by a site safety meeting.
4. Identify and stake all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner or field team prior to soil sampling.

8.3 Representative Sample Collection

The objective of representative sampling is to ensure that a sample or group of samples adequately reflect site conditions.

8.3.1 Sampling Approaches

It is important to select an appropriate sampling approach for accurate characterization of site conditions. Each approach is defined below. Table 8-1 summarizes the following sampling approaches and ranks them from most to least suitable based on the sampling objective.



Table 8-1

REPRESENTATIVE SAMPLING APPROACH COMPARISON

Sampling Objective	Judgmental	Random	Stratified Random	Systematic Grid	Systematic Random	Search	Transect
Establish Threat	1	4	3	2 ^a	3	3	2
Identify Sources	1	4	2	2 ^a	3	2	3
Delineate Extent of Contamination	4	3	3	1 ^b	1	1	1
Evaluate Treatment and Disposal Options	3	3	1	2	2	4	2
Confirm Cleanup	4	1 ^c	3	1 ^b	1	1	1 ^c

1 Preferred approach.

2 Acceptable approach.

3 Moderately acceptable approach.

4 Least acceptable approach.

a Should be used with field analytical screening.

b Preferred only where known trends are present.

c Allows for statistical support of cleanup verification if sampling over entire site.

d May be effective with compositing techniques if site is presumed to be clean.

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8.3.1.1 Judgmental Sampling

Judgmental sampling is based on the subjective selection of sampling locations relative to historical site information, on-site investigation (site walk-over), etc. There is no randomization associated with this sampling approach because samples are collected primarily at areas of suspected highest contaminant concentrations. Therefore, any statistical calculations based on the sampling results would be unfairly biased.

8.3.1.2 Random Sampling

Random sampling involves the arbitrary collection of samples within a defined area. Refer to EPA 1984 and February 1989 for a random number table and guidelines on selecting sample coordinates. The arbitrary selection of sample locations requires each sample location to be chosen independently so that results in all locations within the area of concern have an equal chance of being selected. To facilitate statistical probabilities of contaminant concentration, the area of concern must be homogeneous with respect to the parameters being monitored. Thus, the higher the degree of heterogeneity, the less the random sampling approach will reflect site conditions (see Figure 8-1).

8.3.1.3 Stratified Random Sampling

Stratified random sampling relies primarily on historical information and prior analytical results to divide the area of concern into smaller sampling areas, or "strata." Strata can be defined by several factors, including: sampling depth, contaminant concentration levels, and contaminant source areas. Sampling locations should be selected within a strata using random selection procedures (see Figure 8-2).

8.3.1.4 Systematic Grid Sampling

Systematic grid sampling involves the division of the area of concern into smaller sampling areas using a square or triangular grid. Samples are then collected from the intersection of the grid lines, or "nodes." The origin and direction for placement of the grid should be selected by using an initial random point. The distance between nodes is dependent upon the size of the area of concern and the number of samples to be collected (see Figure 8-3).

8.3.1.5 Systematic Random Sampling

Systematic random sampling involves dividing the area of concern into smaller sampling areas as described in Section 8.3.1.4. Samples are collected within each grid cell using random selection procedures (see Figure 8-4).

8.3.1.6 Biased-Search Sampling

Search sampling utilizes a systematic grid or systematic random sampling approach to define areas where contaminants exceed cleanup standards (i.e., hot spots). The distance between the grid lines and number of samples to be collected are dependent upon the acceptable level of error (i.e., the chance of missing a hot spot). This sampling approach requires that assumptions be made regarding the size, shape, and depth of hot spots (see Figure 8-5).



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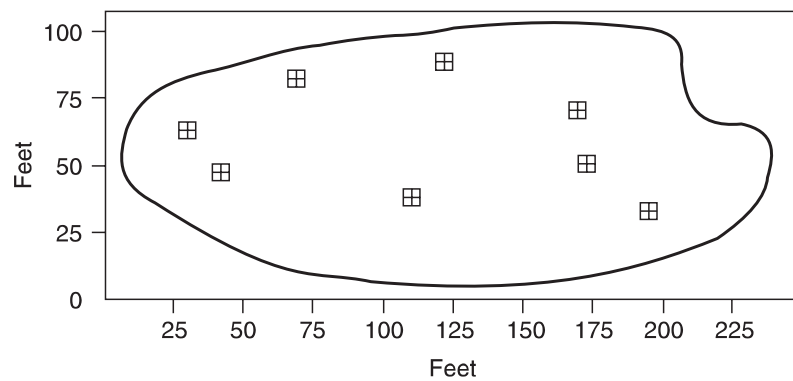


Figure 8-1 RANDOM SAMPLING**

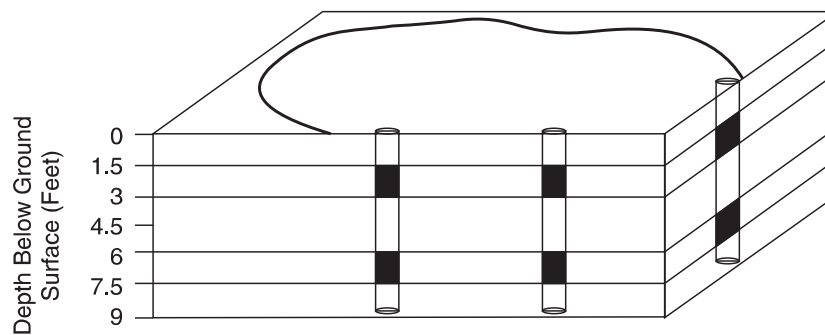


Figure 8-2 STRATIFIED RANDOM SAMPLING

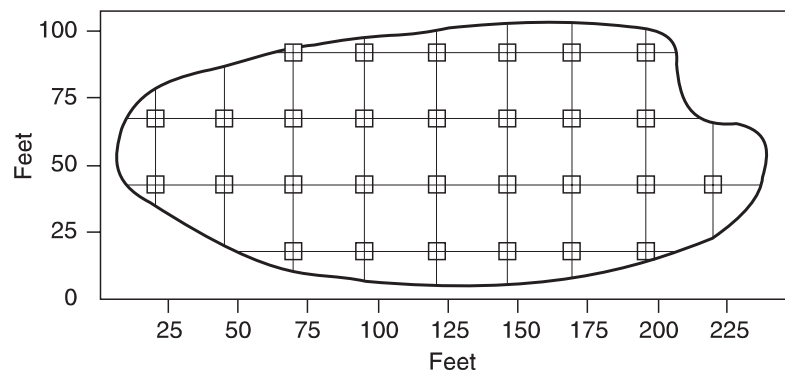


Figure 8-3 SYSTEMATIC GRID SAMPLING**

** After EPA, February 1989

Legend	
—	Sample Area Boundary
⊠	Selected Sample Location
■	Sample Location



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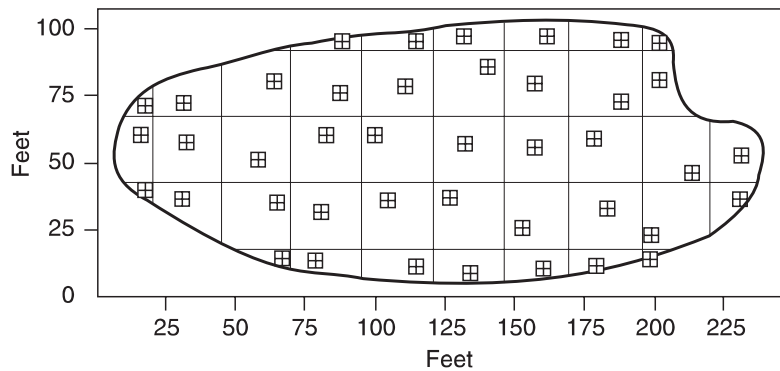


Figure 8-4 SYSTEMATIC RANDOM SAMPLING

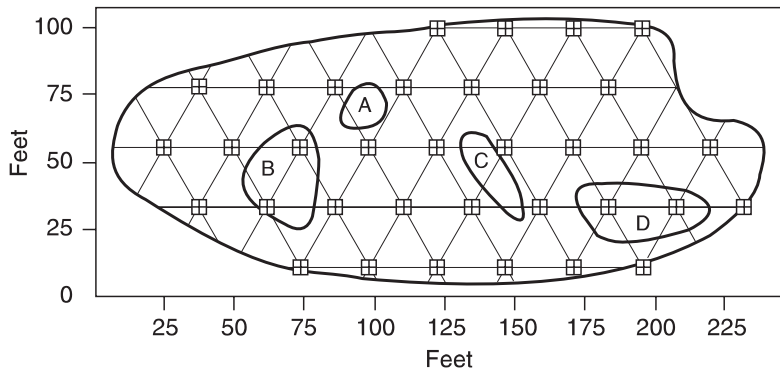


Figure 8-5 SEARCH SAMPLING

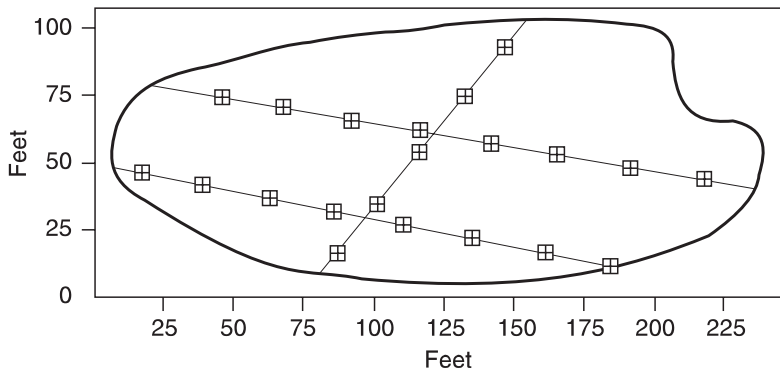
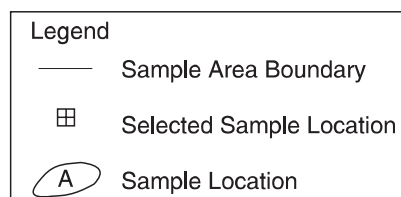


Figure 8-6 TRANSECT SAMPLING

After EPA, February 1989



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8.3.1.7 Transect Sampling

Transect sampling involves establishing one or more transect lines, parallel or nonparallel, across the area of concern. If the lines are parallel, this sampling approach is similar to systematic grid sampling. The advantage of transect sampling over systematic grid sampling is the relative ease of establishing and relocating transect lines as opposed to an entire grid. Samples are collected at regular intervals along the transect line at the surface and/or at a specified depth(s). The distance between the sample locations is determined by the length of the line and the number of samples to be collected (see Figure 8-6).

8.3.2 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, spoons, shovels, and scoops. The surface material can be removed to the required depth with this equipment; stainless-steel or plastic scoops can then be used to collect the sample.

This method can be used in most soil types, but is limited to sampling near-surface areas. Accurate, representative samples can be collected with this procedure, depending on the care and precision demonstrated by the sampling technician. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required (e.g., for volatile organic analysis [VOAs]). A stainless-steel scoop, lab spoon, or plastic spoon will suffice in most other applications. Care should be exercised to avoid the use of devices plated with chrome or other materials, as is common with garden implements such as potting trowels.

Soil samples are collected using the following procedure:

1. Carefully remove the top layer of soil to the desired sample depth with a precleaned spade;
2. Using a precleaned, stainless-steel scoop, spoon, trowel, or plastic spoon, remove and discard the thin layer of soil from the area that came into contact with the shovel;
3. Transfer the sample into an appropriate container using a stainless-steel or plastic lab spoon or equivalent. If composite samples are to be collected, place the soil sample in a stainless-steel or plastic bucket and mix thoroughly to obtain a homogeneous sample representative of the entire sampling interval. Place the soil samples into labeled containers. **(Caution: Never composite VOA samples);**
4. VOA samples should be collected directly from the bottom of the hole before mixing the sample to minimize volatilization of contaminants;
5. Check to ensure that the VOA vial teflon liner is present in the cap, if required. Fill the VOA vial fully to the top to reduce headspace. Secure the cap tightly. The chemical preservation of solids is generally not recommended. Refrigeration is usually the best approach, supplemented by a minimal holding time;
6. Ensure that a sufficient sample size has been collected for the desired analysis, as specified in the Sampling Plan;
7. Decontaminate equipment between samples according to E & E's SOP for Equipment Decontamination (see ENV 3.15); and

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8. Fill in the hole and replace grass turf, if necessary.

QA/QC samples should be collected as specified, according to the Work Plan.

8.3.3 Sampling at Depth with Augers and Thin-Walled Tube Samplers

This system consists of an auger, a series of extensions, a T-handle, and a thin-walled tube. The auger is used to bore a hole to a desired sampling depth and is then withdrawn. The auger tip is then replaced with a tube core sampler, lowered down the borehole, and driven into the soil to the completion depth. The core is then withdrawn and the sample is collected.

Several augers are available, including: bucket type, continuous flight (screw), and posthole augers. Because they provide a large volume of sample in a short time, bucket types are better for direct sample recovery. When continuous-flight augers are used, the sample can be collected directly off the flights, usually at 5-foot intervals. The continuous-flight augers are satisfactory for use when a composite of the complete soil column is desired. Posthole augers have limited utility for sample collection, as they are designed to cut through fibrous, rooted, swampy soil.

The following procedures will be used for collecting soil samples with the hand auger:

1. Attach the auger bit to a drill rod extension, and attach the T-handle to the drill rod.
2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first 3- to 6-inches of surface soil from an area approximately 6 inches in radius around the drilling location.
3. Begin augering, periodically removing and depositing accumulated soils onto a canvas or plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from the boring. When sampling directly from the auger, collect the sample after the auger is removed from boring and proceed to Step 11.
5. A precleaned stainless-steel auger sleeve can also be used to collect a sample. After reaching the desired sampling depth, remove the auger and place the sleeve inside the auger. Collect the sample with the auger. Remove the auger from the boring. The sample will be collected only from the sleeve. The soil from the auger tip should never be used for the sample.
6. Remove auger tip from drill rods and replace with a precleaned thin-walled tube sampler. Install proper cutting tip.
7. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring, as the vibrations may cause the boring walls to collapse.
8. Remove the tube sampler and unscrew the drill rods.
9. Remove the cutting tip and core from the device.



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10. Discard the top of the core (approximately 1 inch), as this represents material collected before penetration of the layer in question. Place the remaining core into the sample container.
11. If required, ensure that a teflon liner is present in the cap. Secure the cap tightly onto the sample container. Place the sample bottle in a plastic bag and put on ice to keep the sample at 4°C.
12. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).
13. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Verify that the chain-of-custody form is correctly and completely filled out.
14. Record the time and date of sample collection, as well as a description of the sample in the field logbook.
15. If another sample is to be collected in the sample hole, but at a greater depth, re-attach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
16. Abandon the hole according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.
17. Decontaminate the sampling equipment as per E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.3.4 Sampling at Depth with a Trier

1. Insert the trier into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample material. Extraction of samples may require tilting of the containers.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. Transfer the sample into a suitable container with the aid of a spatula and brush.
5. If required, ensure that a teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).
6. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).
7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.

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8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.
9. Abandon the hole according to applicable regulations. Generally, shallow holes can simply be backfilled with the removed soil material.
10. Decontaminate sampling equipment as per E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.3.5 Sampling at Depth with a Split-Spoon (Barrel) Sampler

The procedure for split spoon sampling describes the extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be sampled to give a complete soil column, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom the augured hole and the core extraction.

This sampling device may be used to collect information such as soil density. All work should be performed in accordance with ASTM D 1586-84, *Penetration Test and Split Barrel Sampling of Soils*.

1. Assemble the sampler by aligning both sides of the barrel and then screwing the bit on the bottom and the heavier head piece on top. Install a retaining cap in the head piece if necessary.
2. Place the sampler in a perpendicular position on the sample material.
3. Using a sledge hammer or well ring, if available, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result.
4. Record the length of the tube used to penetrate the material being sampled and the number of blows required to obtain this depth.
5. Withdraw the split spoon and open by unscrewing the bit and head. If a split sample is desired, a clean stainless-steel knife should be used to divide the tube contents in half, lengthwise. This sampler is available in 2- and 3.5-inch diameters. The required sample volume may dictate the use of the larger barrel. If needed, stainless-steel or teflon sleeves can be used inside the split spoon. If sleeves removed from the split spoon are capped immediately, volatilization of contaminants can be reduced. When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved 1974).
6. Cap the sample container, place in a double plastic bag and attach the label and custody seal. Record all pertinent data in the field logbook and complete the sample analysis request form and chain-of-custody record before taking the next sample.
7. If required, preserve or place the sample on ice.
8. Follow proper decontamination procedures and deliver samples to the laboratory for analysis.



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8.3.6 Test Pit/Trench Excavation

These relatively large excavations are used to remove sections of soils when detailed examination of soil characteristics (horizontal, structure, color, etc.) are required. It is the least cost-effective sampling method due to the relatively high cost of backhoe operation.

1. Prior to any excavations with a backhoe, it is important to ensure that all sampling locations are clear of utility lines and poles (subsurface as well as above surface).
2. Using the backhoe, a trench is dug to approximately 3 feet in width and approximately 1 foot below the cleared sampling depth. Place removed or excavated soils on canvas or plastic sheets, if necessary. Trenches greater than 4 feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.
3. A shovel is used to remove a 1- to 2-inch layer of soil from the vertical face of the pit where sampling is to be done.
4. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose soil for sampling. Samples are removed and placed in an appropriate container.
5. If required, ensure that a teflon liner is present in the cap. Secure the cap tightly onto the sample container. Samples are handled in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).
6. Carefully and clearly label the container with the appropriate sample tag, addressing all the categories or parameters listed in E & E's SOP for Sample Packaging and Shipping (see ENV 3.16).
7. Use the chain-of-custody form to document the types and numbers of soil samples collected and logged.
8. Record the time and date of sample collection as well as a description of the sample and any associated air monitoring measurements in the field logbook.
9. Abandon the hole according to applicable State regulations. Generally, excavated holes can simply be backfilled with the removed soil material.
10. Decontaminate sampling equipment, including the backhoe bucket, as per E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.4 Sample Preparation

In addition to sampling equipment, representative sample collection includes sample quantity, volume, preservation, and holding time (see Table 8-2). Sample preparation refers to all aspects of sample handling after collection. How a sample is prepared can affect its representativeness. For example, homogenizing can result in a loss of volatiles and is therefore inappropriate when volatile contaminants are the concern.



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Table 8-2

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
SW-846								
VOA ^e	14 days from date sampled	14 days from date sampled	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4 °C (ice in cooler)	Add HCl until pH <2 and cool to 4 °C (ice in cooler)
Semi-VOA (BNAs) ^e	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
PCBs ^{d,e}	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
Pesticides/PCBs ^{d,e}	14 days to extract from date sampled	7 days to extract from date sampled	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
Metals ^c	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Add HNO ₃ until pH <2 and cool to 4 °C (ice in cooler)
Cyanide ^e	14 days from date sampled	14 days from date sampled	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Add NaOH until pH >12 and cool to 4 °C (ice in cooler)
Hexavalent chromium ^a	24 hours from time sampled	24 hours from time sampled	10 g	50 ml	8 oz. glass jar with Teflon-lined cap	125 ml polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
Total Organic Carbon (TOC) ^a	NA	28 days from date sampled	5 g	10 ml	8 oz. glass jar with Teflon-lined cap	125 ml polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Add H ₂ SO ₄ until pH <2 and cool to 4 °C (ice in cooler)

Key at end of table.

Table 8-2

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
Total Organic Halides (TOX) ^{a,e}	NA	7 days from date sampled	100 g	200 ml	8 oz. glass jar with Teflon-lined cap	1 L amber glass bottle	Cool to 4 °C (ice in cooler)	Add H ₂ SO ₄ until pH <2 and cool to 4 °C (ice in cooler)
Total Recoverable Petroleum Hydrocarbons ^e	28 days from date sampled	28 days from date sampled	50 g	1 L	8 oz. glass jar with Teflon-lined cap	1 L amber glass bottle	Cool to 4 °C (ice in cooler)	Add H ₂ SO ₄ until pH <2 and cool to 4 °C (ice in cooler)
USEPA-CLP								
VOA ^e	10 days from date received	10 days from date received	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4 °C (ice in cooler)	Add HCl until pH <2 and cool to 4 °C (ice in cooler)
Semi-VOA (BNAs) ^e	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
PCBs ^{d,e}	10 days to extract from date received	5 days to extract from date received	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
Pesticides/PCBs ^{d,e}	10 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
Metals ^c	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Add HNO ₃ to pH <2 and cool to 4 °C (ice in cooler)
Cyanide ^e	12 days from date received	12 days from date received	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Add NaOH to pH >12 and cool to 4 °C (ice in cooler)
NYSDEC-CLP								
VOA ^{b,e}	7 days from date received	10 days from date received	15 g	One 40 ml vial; no air space	Two 40 ml vials; no air space	Two 40 ml vials; no air space	Cool to 4 °C (ice in cooler)	Add HCl until pH <2 and cool to 4 °C (ice in cooler)
Semi-VOA (BNAs) ^e	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)

Key at end of table.



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Table 8-2

STANDARD SAMPLE HOLDING TIMES, PRESERVATION METHODS, AND VOLUME REQUIREMENTS

Protocol Parameter	Holding Time		Minimum Volume Required		Container Type		Preservation	
	Soil	Water	Soil	Water	Soil	Water	Soil	Water
PCBs ^{d,e}	5 days to extract from date received	5 days to extract from date received	30 g	1 L	4 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
Pesticides/PCBs ^{d,e}	5 days to extract from date received	5 days to extract from date received	30 g	1 L	8 oz. glass jar with Teflon-lined cap	1/2 gallon amber glass bottle	Cool to 4 °C (ice in cooler)	Cool to 4 °C (ice in cooler)
Metals ^c	6 months from date sampled	6 months from date sampled	10 g	300 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Add HNO ₃ to pH <2 and cool to 4 °C (ice in cooler)
Cyanide ^e	12 days from date received	12 days from date received	10 g	100 ml	8 oz. glass jar with Teflon-lined cap	1 L polyethylene bottle with polyethylene-lined cap	Cool to 4 °C (ice in cooler)	Add NaOH to pH >12 and cool to 4 °C (ice in cooler)
EPA Water and Waste								
Total dissolved solids (TDS)	NA	7 days from date sampled	NA	200 ml	NA	1 L polyethylene bottle with polyethylene-lined cap	NA	Cool to 4 °C (ice in cooler)

Note: All sample bottles will be prepared in accordance with EPA bottle-washing procedures. These procedures are incorporated in E & E's Laboratory and Field Personnel Chain-of-Custody Documentation and Quality Assurance/Quality Control Procedures Manual, July 1987.

- ^a Technical requirements for sample holding times have been established for water matrices only. However, they are also suggested for use as guidelines in evaluating soil data.
- ^b Holding time for GC/MS analysis is 7 days if samples are not preserved.
- ^c Maximum holding time for mercury is 28 days from time sampled.
- ^d If one container has already been collected for PCBs analysis, then only one additional container need be collected for extractable organics, BNAs, or pesticides/PCBs analysis.
- ^e Extra containers required for MS/MSD.

Key:
NA = Not applicable.



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8.4.1 Sample Quantity and Volume

The volume and number of samples necessary for site characterization will vary according to the budget, project schedule, and sampling approach.

8.4.2 Sample Preservation and Holding Time

Sample preservation and holding times are as discussed in Section 4.

8.4.3 Removing Extraneous Material

Discard materials in a sample that are not relevant for site or sample characterization (e.g., glass, rocks, and leaves), since their presence may introduce an error in analytical procedures.

8.4.4 Homogenizing Samples

Homogenizing is the mixing of a sample to provide a uniform distribution of the contaminants. Proper homogenization ensures that the containerized samples are representative of the total soil sample collected. All samples to be composited or split should be homogenized after all aliquots have been combined. Do not homogenize samples for volatile compound analysis.

8.4.5 Compositing Samples

Compositing is the process of physically combining and homogenizing several individual soil aliquots of the same volume or weight. Compositing samples provides an average concentration of contaminants over a certain number of sampling points. Compositing dilutes high concentration aliquots; therefore, detection limits should be reduced accordingly. If the composite area is heterogeneous in concentration and its composite value is to be compared to a particular action-level, then that action-level must be divided by the total number of aliquots making up the composite for accurate determination of the detection limit.

8.4.6 Splitting Samples

Splitting samples (after preparation) is performed when multiple portions of the same samples are required to be analyzed separately. Fill the sample containers simultaneously with alternate spoonfuls of the homogenized sample (see Figure 8-7).

8.5 Post-Operations

8.5.1 Field

Decontaminate all equipment according to E & E's SOP for Equipment Decontamination (see ENV 3.15).

8.5.2 Office

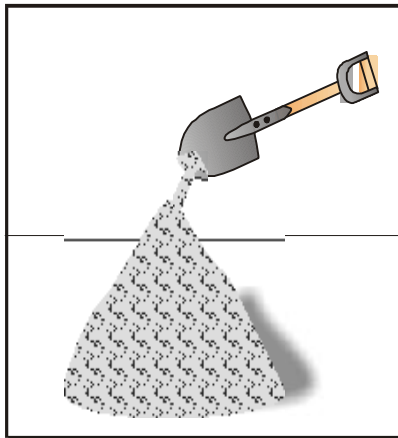
Organize field notes into a report format and transfer logging information to appropriate forms.



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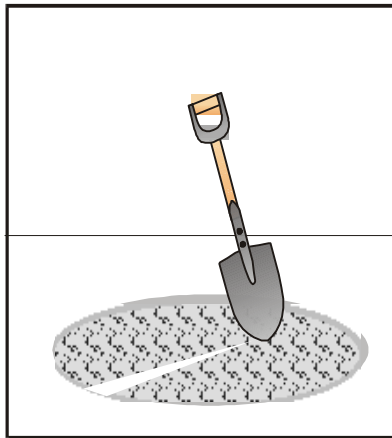
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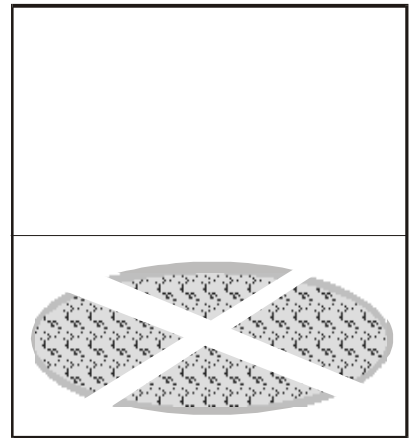
Step 1:

- Cone Sample on hard, clean surface
- Mix by forming new cone



Step 2:

- Quarter after flattening cone



Step 3:

- Divide sample into quarters

Step 4:

- Remix opposite quarters
- Reform cone
- Repeat a minimum of 5 times

After: ASTM Standard C702-87

Figure 8-7 Quartering to Homogenize and Split Samples



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9. Calculations

There are no specific calculations required for these procedures.

10. Quality Assurance/Quality Control

The objective of QA/QC is to identify and implement methodologies that limit the introduction of error into sampling and analytical procedures.

10.1 Sampling Documentation

10.1.1 Soil Sample Label

All soil samples shall be documented in accordance with E & E's SOP for Sample Packaging and Shipping (see ENV 3.16). The soil sample label is filled out prior to collecting the sample and should contain the following:

1. Site name or identification.
2. Sample location and identifier.
3. Date samples were collected in a day, month, year format (e.g., 03 Jan 88 for January 3, 1988).
4. Time of sample collection, using 24-hour clock in the hours: minutes format.
5. Sample depth interval. Units used for depths should be in feet and 10ths of feet.
6. Preservatives used, if any.
7. Analysis required.
8. Sampling personnel.
9. Comments and other relevant observations (e.g., color, odor, sample technique).

10.1.2 Logbook

A bound field notebook will be maintained by field personnel to record daily activities, including sample collection and tracking information. A separate entry will be made for each sample collected. These entries should include information from the sample label and a complete physical description of the soil sample, including texture, color (including notation of soil mottling), consistency, moisture content, cementation, and structure.

10.1.3 Chain-of-Custody

Use the chain-of-custody form to document the types and numbers of soil samples collected and logged. Refer to E & E's SOP for Sample Packaging and Shipping (see ENV 3.16) for directions on filling out this form.



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10.2 Sampling Design

1. Sampling situations vary widely; thus, no universal sampling procedure can be recommended. However, a sampling plan should be implemented before any sampling operation is attempted, with attention paid to contaminant type and potential concentration variations.
2. Any of the sampling methods described here should allow a representative soil sample to be obtained, if the sampling plan is properly designed.
3. Consideration must also be given to the collection of a sample representative of all horizons present in the soil. Selection of the proper sampler will facilitate this procedure.
4. A stringent QA project plan should be outlined before any sampling operation is attempted. This should include, but not be limited to, properly cleaned samplers and sample containers, appropriate sample collection procedures, chain-of-custody procedures, and QA/QC samples.

11. Data Validation

The data generated will be reviewed according to the QA/QC considerations that are identified in Section 10.

11.1 Quality Assurance/Quality Control Samples

QA/QC samples are used to identify error due to sampling and/or analytical methodologies and chain-of-custody procedures.

11.1.1 Field Duplicates (Replicates)

Field duplicates are collected from one location and treated as separate samples throughout the sample handling and analytical processes. These samples are used to assess total error for critical samples with contaminant concentrations near the action level.

11.1.2 Collocated Samples

Collocated samples are generally collected 1.5 to 3.0 feet away from selected field samples to determine both local soil and contaminant variations on site. These samples are used to evaluate site variation within the immediate vicinity of sample collection.

11.1.3 Background Samples

Background or "clean" samples are collected from an area upgradient from the contamination area and representative of the typical conditions. These samples provide a standard for comparison of on-site contaminant concentration levels.

11.1.4 Rinsate (Equipment) Blanks

Rinsate blanks are collected by pouring analyte-free water (i.e., laboratory de-ionized water) on decontaminated sampling equipment to test for residual contamination. These samples are used to assess potential cross contamination due to improper decontamination procedures.



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11.1.5 Performance Evaluation Samples

Performance evaluation samples are generally prepared by a third party, using a quantity of analyte(s) known to the preparer but unknown to the laboratory. The percentage of analyte(s) identified in the sample is used to evaluate laboratory procedural error.

11.1.6 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

MS/MSD samples are spiked in the laboratory with a known quantity of analyte(s) to confirm percent recoveries. They are primarily used to check sample matrix interferences.

11.1.7 Field Blanks

Field blanks are prepared in the field with certified clean sand, soil, or water. These samples are used to evaluate contamination error associated with sampling methodology and laboratory procedures.

11.1.8 Trip Blanks

Trip blanks are prepared prior to going into the field using certified clean sand, soil, or water. These samples are used to assess error associated with sampling methodology and analytical procedures for volatile organics.

12. Health and Safety

12.1 Hazards Associated With On-Site Contaminants

Depending on site-specific contaminants, various protective programs must be implemented prior to soil sampling. The site Health and Safety Plan should be reviewed with specific emphasis placed on a protection program planned for direct-contact tasks. Standard safe operating practices should be followed, including minimization of contact with potential contaminants in both the vapor phase and solid matrix by using both respirators and disposable clothing.

Use appropriate safe work practices for the type of contaminant expected (or determined from previous sampling efforts):

- Particulate or Metals Contaminants
 - Avoid skin contact with, and ingestion of, soils and dusts.
 - Use protective gloves.
- Volatile Organic Contaminants
 - Pre-survey the site with an HNu 101 or OVA 128 prior to taking soil samples.
 - If monitoring results indicate organic constituents, sampling activities may be conducted in Level C protection. At a minimum, skin protection will be afforded by disposable protective clothing.



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A. Sampling Augers

A. Sampling Augers



(a)
Ship Auger



(b)
Closed-Spiral Auger



(c)
Open-Spiral Auger

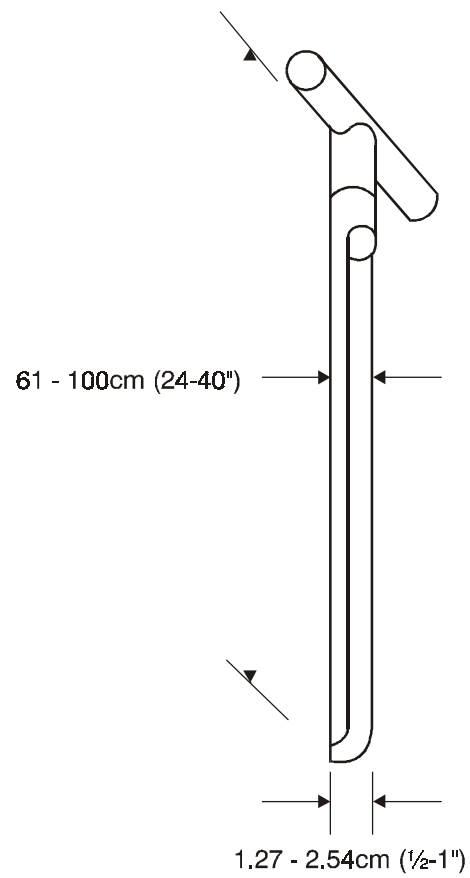


(d)
Iwan Auger



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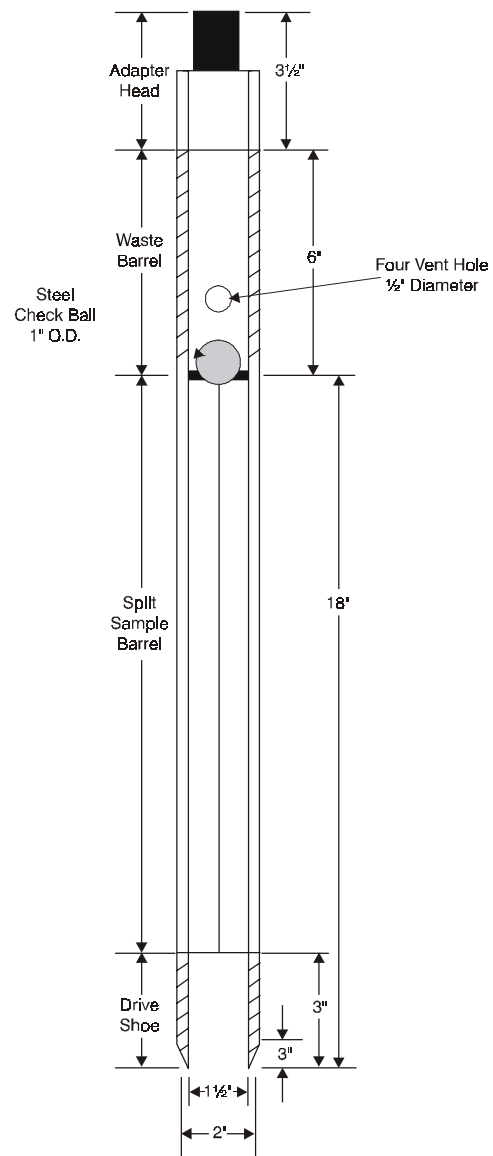
B. Sampling Trier





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C. Split-Spoon Sampler



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March 1998

STANDARD OPERATING PROCEDURE

HAZARDOUS WASTE SITE ENTRY AND EGRESS

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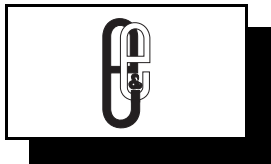
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1. Introduction

Key elements in any hazardous waste site investigation is entry and egress into the site. Safe and proper entry/egress requires a careful and coordinated team effort.

The entry team must conduct and complete reconnaissance of the site, as well as undertake other tasks in unfamiliar surroundings, in a variety of situations and with unknown hazards. The goals are to complete the tasks while maintaining and protecting the health and safety of Ecology and Environment, Inc. (E & E), and subcontractor personnel.

E & E has established Standard Operating Procedures (SOPs) to serve as training and guidance tools during the planning and implementation of all phases of a site investigation. Of primary concern are the health and safety of personnel. This is organized under the Corporate Health and Safety Program and blueprinted in the Corporate Health and Safety Plan for Toxic and Hazardous Substances. Other phases of investigations are addressed in other SOPs; thus, those SOPs should be carefully reviewed prior to a site investigation.

This document presents site entry/egress procedures and guidelines for all E & E personnel working at hazardous waste sites.

1.1 Objectives

The primary objective of the SOP for Hazardous Waste Site Entry and Egress is to provide operating guidelines and establish procedures for the entry/egress and inspection of hazardous waste sites. It encompasses preplanning, site safety considerations, entry/egress procedures, on-site guidelines, site egress, and decontamination procedures. This document is not designed to present site-specific procedures, but describes procedures applicable to a wide variety of hazardous waste sites.

1.2 Scope and Limitations

The procedures outlined in this SOP are applicable to all E & E personnel who participate in the inspection or investigation of sites where chemical or biological contamination is known or suspected.

The provisions of this document are not binding to non-E & E personnel, unless otherwise specified under contractual agreement. However, non-E & E personnel are encouraged to follow these procedures.

This document is applicable only to the conditions and limitations specified within and does not address operations at radioactive or asbestos sites, drum-opening operations, or where confined-space entries are anticipated. For asbestos inspection, assessment, or abatement oversight projects, please refer to *E & E Corporate Guidelines for Asbestos Inspection/Assessment, Laboratory Analysis, and Abatement Oversight* (October 1987). Radiation or confined-space work requires pre-approval from E & E's Health and Safety Group in Buffalo. E & E's confined space entry SOP



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should be followed and the included permits completed whenever site activities involve entry into confined space.

2. Policies and Documentation

2.1 Policies

The health and safety of site inspection personnel have precedence over all other considerations in dealing with hazardous materials. When inspection personnel must be exposed to hazardous substances, every effort should be made to ensure that exposure concentrations are as low as possible. E & E has established specific health and safety policies and guidelines in the E & E Health and Safety Plan for Toxic and Hazardous Substances.

Entry into a known or suspected contaminated waste site will be conducted only when such entry is required to gather information not obtainable by any non-entry means. The duration of time spent on site will be kept as short as possible. Entry into any hazardous waste site is expressly forbidden unless conducted as part of a work assignment.

The regional manager/supervisor and/or the designated assistant will appoint a site safety officer (SSO) for each on-site inspection or investigation where hazardous substance contamination is suspected.

During development of the site-specific health and safety plan (SHASP), decisions will be made by the project team with the Health and Safety Group support regarding:

- Potential hazards present;
- Type of protective clothing to be worn;
- Type of respiratory protection to be worn;
- Type of decontamination procedures to be used;
- Distance to the exclusion area boundary; and
- Location of the command post (CP), personnel decontamination station (PDS), and contamination control line (CCL).

The project team leader (PTL) will assume full responsibility for the action of E & E personnel only. The PTL will assume no responsibility for the action of noncontractor personnel. Noncontractor personnel entering the hazardous waste site will participate in site briefings and be advised of the hazards present and safety precautions deemed necessary. All companies participating in work requiring site entry must have developed their own SHASP prior to such entries. If noncontractor personnel choose to enter the hazardous waste site despite the PTL's advice, official



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written notification will be made in the daily log and the regional manager/supervisor will be advised of the situation. The project manager (PM) will also be advised of the situation. If the actions of non-E & E personnel result in a threat to the welfare of E & E personnel, the PTL can choose to cease on-site operations until the situation is resolved.

2.2 Documentation

Before site entry, documentation should be completed to address the legal, health, and safety issues of site access. Legal issues concerning E & E's right to enter the site are resolved by obtaining permission to access the property(ies) that contain the site. This permission may be granted by the property owner(s) or by appropriate judicial authorities. Documentation of permission, in the form of a court order or written authorization, should be available for disclosure at the site during the investigation.

Written permission to dispose of investigation-derived waste on site must be obtained prior to site work. If this cannot be obtained, arrangements must be made for off-site disposal.

The PTL, PM, and SSO will be responsible for maintaining all records, logs, and reports required for, or generated by, the on-site activities (refer to E & E's SOP for *Field Activities Logbooks* [DOC 2.1]). Records will be kept of the following:

- A log of all information (e.g., calibrations) and all data from personnel monitoring devices and instruments (e.g., dosimeters and personnel air sampling devices);
- An on-site activity log (to be updated hourly) to record the weather conditions, progress of each activity, field data from each sample collected, tracking numbers, and other information for all samples; and
- An on-site safety log of all personnel, safety activities (e.g., monitoring activities and results), site entries, personal protective equipment (PPE) usage, decontamination procedures, deviations from the original safety plan, protective steps taken, special problems encountered, and solution measures taken.

In addition, photographic documentation of site conditions may be used during investigations and can prove to be indispensable for later use in site evaluations, reports, or legal proceedings. All information concerning each photograph must be recorded in the field logbook or a separate photographic log.



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3. Field Operations

3.1 Site Safety Considerations

Assurance of a safe investigation hinges on proper planning and implementation. The following are four fundamental site safety components:

- Thorough background search for site characterization;
- Development and approval of a comprehensive SHASP and work plan;
- Implementation of the approved SHASP through meetings and training; and
- Use of safe work practices by adhering to general safety rules and requirements.

3.1.1 Background Search for Site Characterization

The background search occurs during the planning stages of the projects and is inherently linked to the formation of the SHASP and work plan (see Section 3.1.2). Staff conduct a review of all background material and available data, focusing on the past history of the site, the materials used or disposed of at the site, data and information from previous investigations, past site conditions, litigation-related documentation, and the present activity at, and condition of, the site. This information is available from a variety of sources:

- Client records,
- Aerial photographs,
- Federal, state, or local agency files,
- National or regional data bases, and
- Wire service searches for press items.

Because sites vary, E & E has developed three categories, or types of sites, for planning purposes. Levels of personnel and respiratory protection are selected based on site characteristics:



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Site Type I

A Type I site is an apparent "no-problem" site. All background information indicates that no hazard is associated with this site. If the correct response to all of the items listed below can be obtained, then E & E personnel may conduct the investigation in Level D protection.

- Attempt to complete the investigation with perimeter reconnaissance.
- If there is a need to enter, do so (Level D) only if the following apply:
 - The task is limited to a brief visual inspection;
 - No containers of unknown or known hazardous substances are visible;
 - There are no unknown odors or known odors of hazardous substances;
 - The site has good natural air circulation;
 - There is visible or historic evidence that humans have accessed the site with no ill health effects; and
 - Dead animals or vegetation are not visible.

Site Type II

A Type II site is an industrial workplace site. Because there are workers already present, active industrial sites do not generally pose an imminent danger. However, team members must nonetheless conduct extensive research to determine whether the safety precautions in effect at the industrial workplace site are adequate for E & E personnel. The review process is outlined below:

- Review the site history and establish the type of workplace;
- Inventory the workplace chemical hazards;
- Establish applicable industry standards (consult regional and/or corporate Health and Safety Plan);
- If the facility's ongoing health and safety program is acceptable, and the investigative task presents no additional risk, use the facility program as a basis for E & E's SHASP.

If the review process indicates that the existing level of protection is adequate, then the team may enter the site in the same level of protection as the industrial workplace personnel. If the review



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process indicates that a higher level of protection is required, these findings should first be discussed with the site authorities. Team members should not appear at a site in a higher level of protection without first informing the site managers.

Site Type III

A Type III site is a hazardous waste site. The review process for a hazardous waste site is listed below:

- Evaluate the need for Level A—Emergency Action;
- Determine whether splash protection is needed for initial entry;

At the time of the preparation of this SOP revision, E & E did not maintain Level A capabilities in all regional offices. Level A work must be approved by Corporate Health and Safety at headquarters.

- Conduct initial entry;
- Determine whether an apparent respiratory hazard exists:
 - **IF YES:** Continue the investigation in a self-contained breathing apparatus (SCBA) and any necessary splash protection;
 - **IF NO:** Continue the investigation without respiratory protection;
- Continue work and conduct ambient monitoring and re-evaluation of personal protection needs.

For a hazardous waste site, the need for higher levels of protection should be evaluated against the potential of skin contact and inhalation. To assist in this evaluation, E & E has developed decision guidance matrices for assigning personnel Levels of Protection (LOP) at a Type III site (see Figure 1). These decision matrices require simple "yes" or "no" answers to achieve succeeding levels of decision.

After determining the proper clothing for site entry, teams must determine whether an apparent respiratory hazard exists. No apparent respiratory hazard exists if:



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Figure 1 Decision Guidance for Assigning LOP at Type III Sites



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- No drums, lagoons, or other containers of unknown contents exist in close proximity to the work area;
- The site has good air circulation;
- No ill health effects are associated with the site;
- The investigation does not require excavation, drilling, or drum opening;
- The historical review gives a general idea of chemicals present.

If no respiratory hazard exists, an initial entry survey should then be conducted. If no background information is available to determine respiratory hazard, the SSO should require use of respiratory protection for site entry.

3.1.2 SHASP and Work Plan Development

Many health and safety concerns associated with the inspection or investigation of a hazardous waste site are related to the nature of the site and the extent of the work to be performed. Consequently, the SHASP is a site-specific document that lists the tasks of the on-site work and describes the health and safety procedures and protocols that will be followed for each task.

The SHASP provides a written outline of the steps to be followed and provides a checklist for preparing for site entry. The SHASP identifies and organizes the available data and provides a logical, coherent, and workable plan. The SHASP must be reviewed by designated staff who are able to identify inadequacies before approving the SHASP.

The SHASP must include site background information, safety information, and instructions and procedures for both routine and emergency situations to ensure that the risk of accident or injury is minimized. The SHASP should also allow for contingencies that will permit adaptations, at the discretion of the PM and the SSO, as new information is gathered and/or field conditions fluctuate. Finally, the SHASP must be presented in a concise written format so that it is available to team members as a source of site-specific safety and health information to team members.

Under certain conditions, the SSP may be subject to modifications by the PTL and SSO on site. If the danger appears to be greater than originally anticipated, the SSO has the option of immediately withdrawing the team or increasing the level of protection. If the danger was originally overestimated, the SSO may allow the site to be examined at a lower level of protection, provided that the SHASP describes the rationale and the specific protection level to which the site can be downgraded.

When site conditions warrant modification to the operating procedure or the level of personal protection required, the changes and conditions should be thoroughly documented. If the modification results in significant changes, such as downgrading to a lower level of protection, the regional safety coordinator (RSC) or headquarters safety staff must be informed. The SSO must present the rationale for making the changes and provide sufficient monitoring data to justify the reduction in the level of protection. Team members will not use a level of protection less than that



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required in the SHASP for each specific exclusion area or sub-area until that decision has been approved by the SSO.

The work plan, like the SHASP, includes a written outline of the work to be performed on site and the methods to be used to complete the investigative tasks. Because it is peer-reviewed, inadequacies or unsafe procedures should be identified and modified. In many cases, the work plan is produced as part of the project proposal, or for review by the client.

A work plan should contain details of the site investigation, including:

- Initial entry and characterization;
- Inventory of site materials and conditions;
- Sampling plan and analysis required;
- Geophysical explorations;
- Groundwater monitoring well installation;
- Decontamination methods; and
- Disposal of site-derived materials (e.g., drill cuttings, decontamination fluids, disposable protective wear).

3.1.3 Implementation of SHASP Through Meetings and Training

Before on-site activities are initiated, the SSO, to the degree necessary (based on the site hazard level as determined by the PM), will inform local medical and emergency authorities of planned activities and special treatment or response needs in the event of injury and/or exposure.

Before site entry, the SSO will conduct as many meetings or training sessions as necessary to ensure that all personnel who enter the site have a thorough understanding of health, safety, and operational aspects of on-site activities. Issues to be addressed during these meetings and training sessions may include, but are not limited to, the following:

- Potential chemical and physical hazards associated with the site;
- Safety procedures (including a pre-planned route to the designated emergency medical facility), specific hazards associated with on-site contaminants (as presented in the Hazard Evaluation of Chemicals worksheet), and team response organization and responsibilities in the event of an injury or contamination incident;
- Monitoring instrument operation and monitoring frequency requirements;



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- The level(s) of personnel and respiratory protection required in each phase of planned on-site activity;
- Protective equipment fit and operation;
- Heat/cold stress prevention;
- Decontamination requirements and procedures;
- Site control procedures, including the designation of work and exclusion zones, and site entry and exit procedures; and
- Contingency plans to protect the public and the environment in the event of an emergency.

All team members (including subcontractors and supervisory personnel) will sign the On-Site Safety Meeting Form in the SSO's copy of the SHASP at the end of each safety meeting. By signing the form, each team member indicates that the on-site hazards and site safety procedures and protocols have been explained by the SHASP to his or her understanding. The form must be signed before site entry.

3.1.4 General Safety Rules and Requirements

Team members must be aware of their responsibility to follow the general on-site safety practices and regulations specified in E & E's Health and Safety Plan. These include the following:

- There will be no eating, drinking, smoking, or use of chewing tobacco in the exclusion area or the contamination reduction area.
- All personnel must pass through the contamination reduction area when entering or exiting an exclusion zone at sites where an exclusion zone has been established.
- All monitoring requirements specified in the SHASP will be carried out by trained personnel with properly maintained and calibrated equipment.
- Emergency equipment (e.g., portable showers, eye washes, fire extinguishers) will be on the hot line side of the contamination reduction area for quick access when such equipment has been included in the SHASP. Eyewash units and fire extinguishers will be verified as being filled and operational.
- Specific emergency equipment requirements for the clean side of the contamination reduction area will be specified in the SHASP.



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- At the end of the workday, all personnel who have worked inside the exclusion zone (including the contamination reduction area) will take a shower either on site (if so equipped), or as soon as possible off site, regardless of perceived exposure possibilities.
- All supplied breathing air shall be certified Grade D (American National Standard, Commodity Specification for Air) or better (e.g., Grade E).
- Where practical, all tools and equipment shall be spark-proof, explosion resistant, Underwriters Laboratory (U.L.) bonded, and grounded. Ground-fault circuit interrupters will be used on power supplies used where water is an exposure hazard.
- Fire extinguishers for use on equipment or small fires will be provided at all sites in a location familiar to all site personnel.
- Trailers shall be fitted with steps and railings at doorways.
- All team members will be aware of emergency response and emergency evacuation responsibilities, procedures, protocols, and signals. Deviations from standard emergency protocols will be specified in the SHASP. Personnel responsibilities will be outlined before site entry. A designated site vehicle should be ready, with ignition keys available, for use in emergencies.
- A first aid kit stocked to handle all first aid situations covered in the Red Cross standard first aid course will be available in an accessible location on site at all times. Additional first aid equipment needs and procedures will be specified in the SHASP, as needed.
- Safety and training meeting(s) will be held before site entry and, if necessary, each morning to cover or review all site-specific safety issues. The SSO or PM will log the topics and attendees daily. The command post files should include an SHASP with addenda and maps to hospitals, sealed copies of team Health Summary Reports (HSRs), and logbooks/safety meeting briefing sheets.

Additional site safety procedures are presented below:

- Observe the buddy system;
- Plan your actions—be deliberate;



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- Maintain contact with the safety officer;
- Practice contamination avoidance:
 - Do not sit or kneel on the ground.
 - Protect monitoring equipment and do not place it on the ground.
 - Avoid obvious contamination.
- Do not climb over drums or obstacles;
- Follow predesignated routes;
- Minimize time in the hot zone;
- Follow monitoring procedures according to the SHASP;
- Monitor weather conditions for:
 - Heat stress;
 - Cold (frostbite);
 - Electrical storms; and
 - Wind direction.

3.1.5 Radiological Hazards

Besides physical, chemical, and biological hazards, radiological hazards are possible at known or suspected contaminated waste sites. The inspection team must always be alert to the potential presence of radiological hazards that may represent a threat to health and safety.

If the historical background study does not indicate that radiological materials are present and the initial entry survey shows radiation above the background level, all E & E personnel shall leave the site and the SSO will notify the E & E RSC and/or the Radiation Group at the Buffalo corporate headquarters.

3.2 Organization of the Field Investigation Area

The layout of the field investigation area can be difficult to define. Distances between each of the various components at the field site will vary depending on the weather, terrain, location, and types of contaminants on site. The knowledge acquired through planning, together with an off-site



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reconnaissance around the perimeter of the suspected site, generally will provide the information needed to establish a rough boundary for the site. See Figure 2 for standard organization of the field investigation area.

When establishing the organization and layout of the area, the two main objectives are to minimize the potential for exposure, and to restrict the spread of contaminated materials outside of the hazardous waste site.

3.2.1 Exclusion Area (Control Area)

The first priority in approaching the area of an uncontrolled chemical or biological waste site is to establish an exclusion, or control, area. This area acts as a buffer zone between the known or suspected contaminated area and the clean area. Initially, it is always assumed that the surface within the exclusion area is contaminated. The size of the exclusion area will vary depending on the nature of the site. The factors considered in establishing the exclusion area are:

- Nature and toxicity of the site contaminants,
- Explosive potential,
- Meteorological condition,
- Topography,
- Concern for the unprotected public, and
- Emergency egress routes from the site.

Safety is the primary consideration in establishing the exclusion area boundary. After an initial assessment of the site has been made by the entry party, the size of this area may be reduced or increased accordingly.



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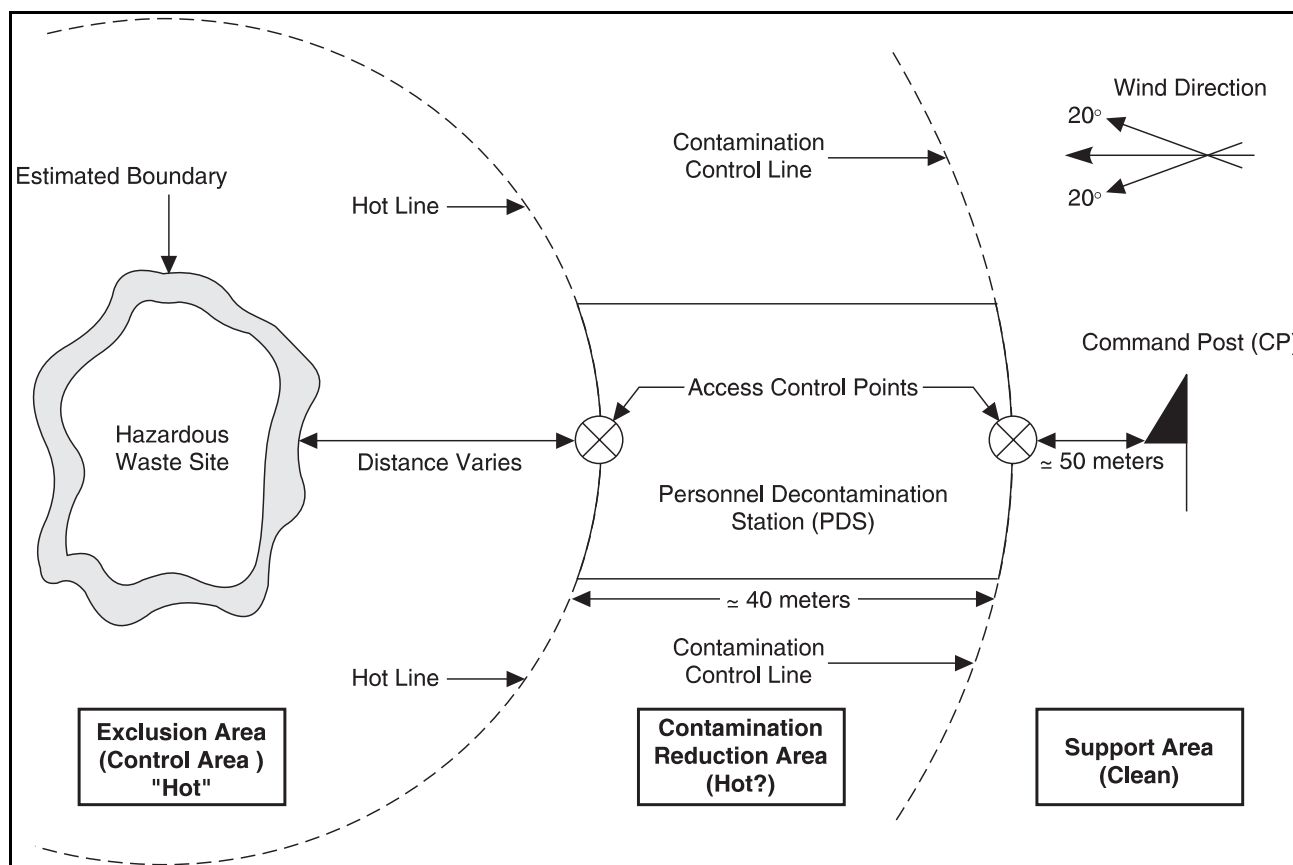


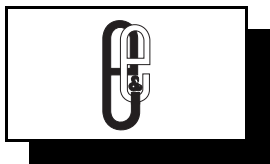
Figure 2 Organization of the Field Investigation Area

3.2.2 Hot Line

The hot line is located on the outer exclusion area boundary. This line is an arbitrary line set at an upwind distance from the hazardous area or spilled chemical material in an effort to control the spread of contamination, afford protection for the decontamination personnel, and limit access to the area. This line should be visually obvious and will be adjusted according to individual site circumstances and hazards. An access control point on the hot line is established through which all personnel are to enter and egress the exclusion area. Personnel or equipment crossing the hot line into the hot zone should do so only when properly protected according to the hazards associated with the site. The hot line can be adjusted in response to additional information learned about the site.

3.2.3 Contamination Reduction Area

The contamination reduction area lies between the hot line and the contamination control line. In this area, a deliberate attempt is made to control and reduce contamination picked up by personnel returning from the exclusion area through the initiation of decontamination operations



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called the Personnel Decontamination Station (PDS). Operations within the PDS are discussed in Section 3.7.

3.2.4 Contamination Control Line

The contamination control line, which is the outer boundary of the contamination reduction area, separates the possibly contaminated area from the clean area. Entry from the clean support area to the contamination reduction area is always made through an access control point on the contamination control line. No person may cross this line unless he or she has the proper level of protection.

3.2.5 Support Area (Administrative)

The support area is always established in a clean area. It contains the command post (CP) and other support elements. The CP is the center of command and control for the entire operation; it is always upwind of the contamination area and, if possible, on high ground to provide visual observation of the entire area.

3.2.6 Wind Direction

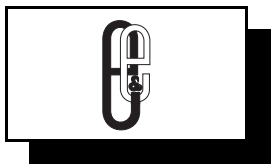
If at all possible, the CP should be located upwind of the hazard area. If terrain or water will not accommodate this arrangement, a crosswind is acceptable. If the CP must be located downwind of the site, all personnel may have to don appropriate breathing apparatus. Significant variations in normal prevailing winds can be monitored through maintaining on-site weather status reports and use of local weather reports for daily planning and updating.

3.3 Organization for Work

The personnel requirements for conducting operations on a hazardous substances site depend on site conditions. A five-member team is shown as an example in Figure 3.

3.3.1 Command and Control

Command and control of the work site is exercised from the CP, which is manned by a CP supervisor or team leader. Ideally, the CP will maintain radio contact with other team personnel and, by radio, will direct their actions. The CP supervisor also maintains contact with the PM and other personnel not on site. In a five-member team, only one team member (usually the team leader) will remain in the CP.



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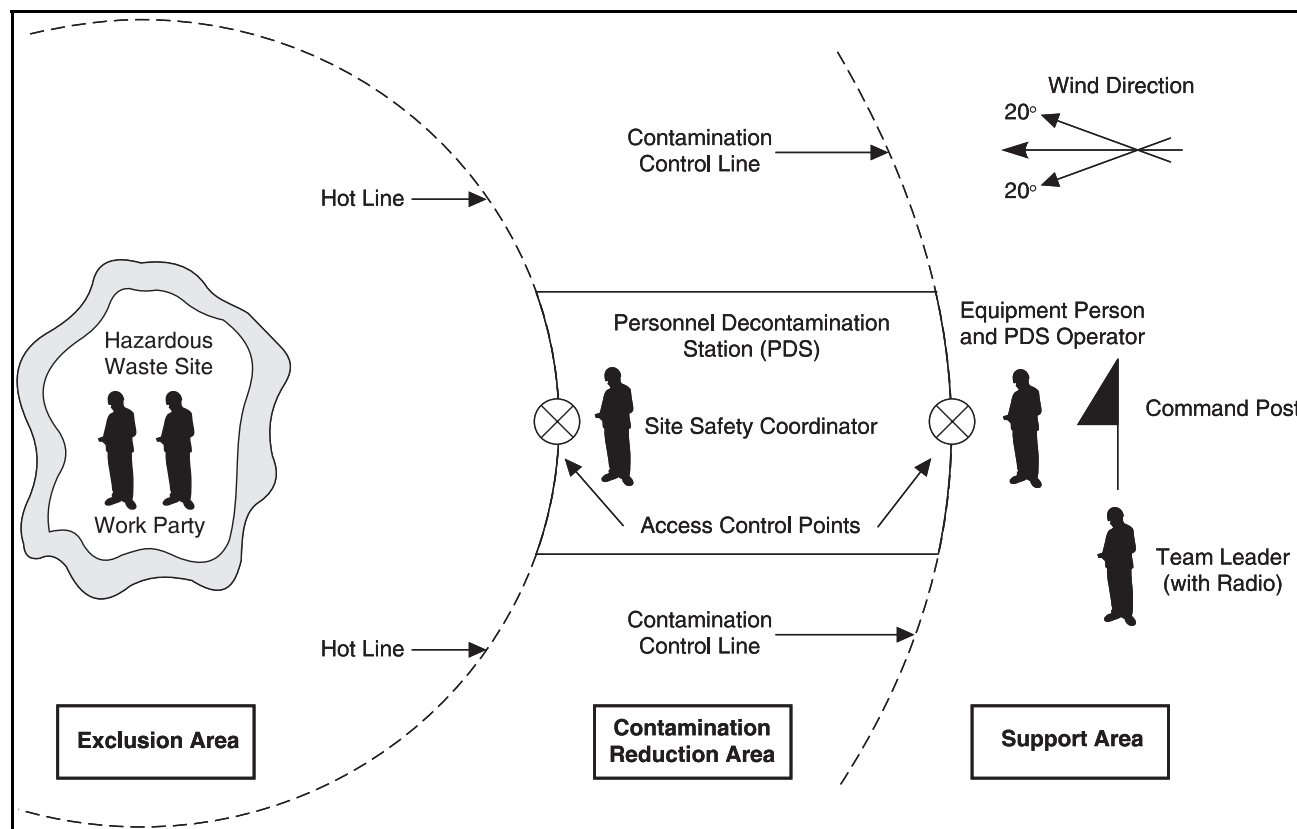


Figure 3 Organization of Work (Five-Member Team)

3.3.2 PDS Operator

At least one team member will be designated as the PDS operator. This team member processes work teams into the hazard site and decontaminates personnel returning from the hazard site. He or she is also responsible for equipment checkout and refilling of SCBA tanks. In a five-person team, the safety officer must help the PDS operator during processing of team members in and out of the PDS.

3.3.3 Safety Officer

The safety officer is positioned on the hot line, where he or she can observe the actions of work parties downrange. A line of site between the safety officer and sampling team should be maintained at all times. The safety officer directs withdrawal of the work parties if, in his or her judgment, actions are being performed that may be unsafe. The safety officer is the primary advisor to the team leader on all matters relating to safety.



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3.3.4 Work Party (Sampling Team)

The final element of a five-member team is the work party. This two-person team performs the necessary tasks called for in the site investigation (e.g., sampling) and plays the following roles:

- Initial entry party—if it is the first team on site. It serves as a reconnaissance team, gathering information about the site.
- Work party—if performing general work or making general observations.
- Sampling team—when sample collection is the sole purpose for being on site.

The work party will never consist of less than two individuals, but may be larger, if required, to accomplish the work directed.

3.4 Air Monitoring and Characterization Equipment

Specialized equipment is used to measure and characterize air contaminants on a hazardous waste site. This information determines the nature and condition of the wastes present, the locations of "hot spots," and the level of worker protection required. This equipment is also used to monitor the site for changes in air contaminant concentrations during investigative work and to screen samples.

Although the instruments are designed for field investigations, they are inherently sensitive to environmental conditions and have defined limitations and interferences, which may cause elevated or suppressed readings. In some cases, a measurement from one instrument may substantiate measurements from another; but in all cases, the information obtained from all instruments should be used to determine the overall condition of the site.

Monitoring instruments should be operated only by trained and experienced personnel who understand the use and limitations of these instruments, and the nature and implications of wastes present at the site. Considerations for developing air monitoring strategies at hazardous waste sites are shown in Table 1. The following subsections describe monitoring equipment.



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Table 1

CONSIDERATIONS FOR AIR TOXIC MONITORING STRATEGIES

Site Conditions	Meteorological Factors	Instrument Factors
<ul style="list-style-type: none"> • Disposal Method (surface impoundment, landfill, drums, landfarm, etc.) • Topography • Surface Area Exposed • Type of Remedial Activity • Level and Frequency of Site Activity • Ambient Air Quality (upwind) • Containment Integrity • Concentration of Contaminant (current) • Age of Site • Predicted Emissions from Remedial Activity • Agents (number, class, compatibility) • Physical/Chemical Properties of Agents • Vehicular Traffic (location and volume) • On-Site Traffic and Equipment Emissions • Intersite Emission Locations 	<ul style="list-style-type: none"> • Wind Speed • Wind Direction • Stability Class • Temperature • Humidity • Solar Loading • Barometric Pressure • Changing Wind Direction and Speed 	<ul style="list-style-type: none"> • Sensitivity/Limits of Detection • Accuracy • Precision • Bias • Specificity/Interferences • Range • Response Time • Service Life • Weight • Portability • Power Requirements • Sorbent Media • Support Equipment (calibration, recorders, sample injection, tools, etc.) • Flow Rate • Sampling Time • Weather Resistance • Ruggedness/Durability



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3.4.1 Explosimeter

The explosimeter is used in combination with an oxygen detector.

- **Description and Use:** The explosimeter determines the level of flammable gases/vapors present in an atmosphere as a percentage of the lower explosive limit (percent LEL) by measuring the change in electrical resistance across a circuit.
- **Operation:** Varies according to make and model. Refer to operations manual for specific instrument.
- **Limitations:** The explosimeter cannot be used in atmospheres in which the oxygen level is below 19.5% or above 25%. Silicanes, silicones, silicates, leaded gasoline vapors, and some caustic/acid vapors can destroy the instrument's sensors. The explosimeter does not indicate whether a given atmosphere is toxic. The instrument must be calibrated daily. It will respond with varying sensitivities to various compounds.
- **Action Levels:**

Refer to Table 2.

3.4.2 Oxygen Detector

The oxygen detector is used in combination with the explosimeter.

- **Description and Use:** The oxygen detector measures the atmospheric oxygen concentration directly by means of a galvanic cell.
- **Operation:** Varies according to make and model. Refer to operations manual for specific instrument.
- **Limitations:** The sensing cell in the oxygen detector has a lifespan of approximately one year. Care must be taken to protect the sensor from immersion in water or damaging blows.
- **Action Levels:** Refer to Table 2.

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Table 2	
HNU PHOTOIONIZER - BRIEF DESCRIPTION OF INSTRUMENT CONTROLS AND FUNCTIONS	
Control	Function
Six-Position Switch	OFF: Shuts off all power and removes DC voltages.
	ON: In any other function position or measuring mode, the electronics are on.
	BATTERY CHECK: Indicates the condition of the battery. If needle position is in lower portion or green battery arc, the instrument should be recharged.
	STANDBY: UV lamp is off but electronics are on. This position will conserve power and extend the useful operating time between recharges of the battery. This position is also utilized to adjust the electronic zero.
	RANGES: 0 to 20, 0 to 200, 0 to 2,000 direct reading ranges available at minimum gain for benzene. More sensitivity is available by adjusting the span potentiometer.
Zero Potentiometer	A 10-turn potentiometer is employed to adjust the zero electronically when the instrument is placed in the standby position with the probe attached. This eliminates the need for a hydrocarbon-free gas.
Span Potentiometer	A 10-turn counting potentiometer is utilized for upscale setting of the meter on calibration gas. Counter-clockwise rotation increases the sensitivity (about 10 times). This control can increase the sensitivity to enable the instrument to directly read nearly any gas the instrument responds to.



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3.4.3 Draeger Tubes

- **Description and Use:** The Draeger tubes measure the concentrations of specific inorganic and organic vapors and gases by creating a discoloration in a detector tube proportional to the amount of material present.
- **Operation:** Refer to operations manual.
- **Limitations:** The pump must be checked frequently for leaks. Response time is relatively slow. Specific tubes must be used for each given compound and are subject to cross-sensitivity. Readings may vary from 5% - 30% depending on compound.
- **Action Levels:** Compound-specific. Consult corporate health and safety group and/or safety plan.

3.4.4 Radiation Instruments

All E & E personnel will wear thermoluminescent dosimeter (TLD) badges during field activities on sites where there is a potential for exposure to radiation. Although these badges create a permanent record of radiation exposure levels, they do not offer "real-time" measurement capability.

- **Description and Use:** The simplest to operate is the Radiation Alert Mini. It detects ionizing radiation and provides visual or audible warnings when preset dose rates are exceeded.

The radiation survey meter is a pulse count rate meter. With the pancake detector probe, it acts as a survey meter for alpha-beta-gamma radiation. Badge or pencil dosimeters give an indication of the total amount of radiation encountered over a period of time. Other instrumentation is available through the radiation group at headquarters.

- **Limitations:** The radiation survey meter must be calibrated frequently and operated only by persons who have been trained in the proper interpretation of its readings. The detector cannot be disconnected while the instrument is in operation.
- **Action Levels:** If greater than background readings occur, evacuate immediately and notify the E & E radiation group at headquarters.



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3.4.5 Photoionization Instruments

- **Description and Use:** Photoionization instruments (PIDs) are used to determine the concentration of select organic and inorganic vapors and gases with an ionization potential (IP) of less than 11.7 electron volts or 10.2 electron volts, depending on the probe in use. Current manufacturers of instruments used by E & E include HNU, Photovac, and Thermoelectron.
- **Operation:** Varies according to make and model. Refer to operations manual for specific instrument.
- **Limitations:** PIDs do not respond to compounds with ionization energies greater than the probe's potential, such as methane (CH₄) or hydrogen cyanide (HCN). The instrument's sensor cannot be immersed in water, and it functions poorly in cold or moist weather. The response will vary with different compounds. The HNU needs to be warmed up prior to use. The HNU can only be used by trained operators. Calibration is required daily.
- **Action Levels:** Refer to Table 2.

3.4.6 Foxboro Organic Vapor Analyzer in the Survey Mode

- **Description and Use:** The OVA provides a continuous readout of the total concentration of organic vapors and gases by using a flame ionization detector (FID).
- **Operation:** The OVA can be used only by specifically trained operators.
- **Limitations:** The OVA needs to be warmed up prior to use and can be used only by specially trained operators. It does not respond to inorganic vapors (most importantly, HCN) but it does respond to methane (CH₄). Due to the small amount of hydrogen required to operate the instrument, shipment can be difficult because of resistance by passenger airlines to accept it as baggage, despite DOT approval.
- **Action Levels:** Refer to Table 2.

3.4.7 Other Equipment

Other equipment that may be used to further evaluate Personal Protection Equipment (PPE) include:

- Particulate monitor (MiniRam),



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- H₂S monitor (hydrogen sulfide),
- HCN monitor (hydrogen cyanide), and
- Portable combination PID/FID (Foxboro TVA 1000).

3.4.8 Initial Site Entry Action Levels

Initial site entry action levels are specifically established in the SHASP. Each E & E employee is responsible for knowing the initial site entry action levels and the appropriate actions to be taken while on a hazardous substance site. Refer to Table 2 for general action levels and appropriate response actions.

3.4.9 Action Level for Radiation

When a reading indicates the presence of any radiation above the background level, E & E employees are to leave the site and report their findings to the SSO or RSC, as appropriate.

3.4.10 Instrument Training

Each E & E employee must be familiar with the equipment used for site characterization. Training in basic instrumentation is part of the 40-hour HAZWOPER training and is covered annually during refresher courses.

3.5 Initial Entry

An initial entry is conducted to confirm whether the historical background review is correct and/or whether site conditions have changed the extent of the hazards. If limited or no information is available, the entry team will determine conditions and hazards.

The team should be linked to the CP by radio communications and will carry all air and radiological monitoring equipment needed to characterize the hazards on site. The members should be constantly alert and watchful for unstable soil, unstable structures or containers, buried or partially buried drums or debris, waste pools or piles, enclosed spaces, protruding objects, or any other dangerous or hazardous conditions. Travel over or through waste should be avoided. Under no circumstances shall any team member climb onto or cross over drums, tanks, or other waste containers. Confined space entries require a preapproved permit to be issued.

Detailed observations are to be recorded in the field logbook, and should include observed hazards, identified container labels, container condition, instrument readings, and apparent spill or waste areas. A Polaroid camera is useful in documenting site conditions, and the photos can be displayed at the CP.

The monitoring instruments must be observed closely. The team should proceed slowly and cautiously to allow for instrument response time, so that the instruments can indicate hazardous



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environments before the team members inadvertently enter one. The team should know the evacuation route from the site should an emergency occur.

A checklist for entry into a hazardous waste site is presented below:

- Actions in Support Area:
 - Work party briefing,
 - Equipment check,
 - Donning of protective clothing,
 - Check out protective ensemble, and
 - Communication check.
- Actions in Contamination Reduction Area:
 - Final communication equipment check, and
 - Enter exclusion zone via access control point.
- Entry to Site:
 - Slow and deliberate,
 - Follow prearranged route,
 - Maintain buddy system,
 - Maintain contact with safety officer,
 - Monitor wind direction, and
 - Conduct ambient air monitoring.

3.6 Egress

Egress from the hazardous site must be carried out as a planned. For example, it is not correct egress procedure to wait for the warning bell to ring on the SCBA oxygen tank before attempting to reach the hot line in time for fresh air supplies. Not only must egress be planned before entry, but it must also be understood that no team member may egress alone, and that no team member may remain on site alone. Operations are always carried out according to the buddy system.

3.7 PDS Procedures (Decontamination)

Before any personnel enter an area where contamination of their clothing, equipment, or person may occur, an adequate decontamination method will be made available. Decontamination methods will conform to the Occupational Safety and Health Administration (OSHA) Guidance Manual for Hazardous Waste Site Activities (National Institute of Occupational Safety and Health [NIOSH], OSHA, USCG, EPA). Disposable clothing will be used whenever possible. All personnel leaving a potentially contaminated area will be appropriately decontaminated in a personnel



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decontamination station (PDS). All equipment, clothing, and cleaning materials will also be appropriately disposed of or decontaminated.

Personnel decontamination has four main functions:

- To ensure that personnel leaving the “hot zone” are properly decontaminated;
- To ensure that all PDS personnel are properly protected during assigned tasks and are decontaminated prior to leaving the site; and
- To leave all contamination at the site.

3.7.1 Location of the PDS

The PDS is set up within the contamination reduction area, which will be located between the contamination control line and the hot line, situated downwind, plus or minus 20-degree wind direction, of the command post (see Figure 4). It is designed to help ensure that all personnel returning from the site have been completely decontaminated before re-entry into the clean support area (see Figure 2).

3.7.2 Level B PDS Organization

Figure 5 is a graphic representation of a typical Level B PDS.

3.7.3 Specifications of the PDS

Although the size of the PDS should be kept to a minimum, it should be accommodating to the needs of the PDS personnel to perform proper decontamination without being contaminated by splashes, spills, or accidental contact with contaminated personnel.

Each PDS is designed individually depending on the following:

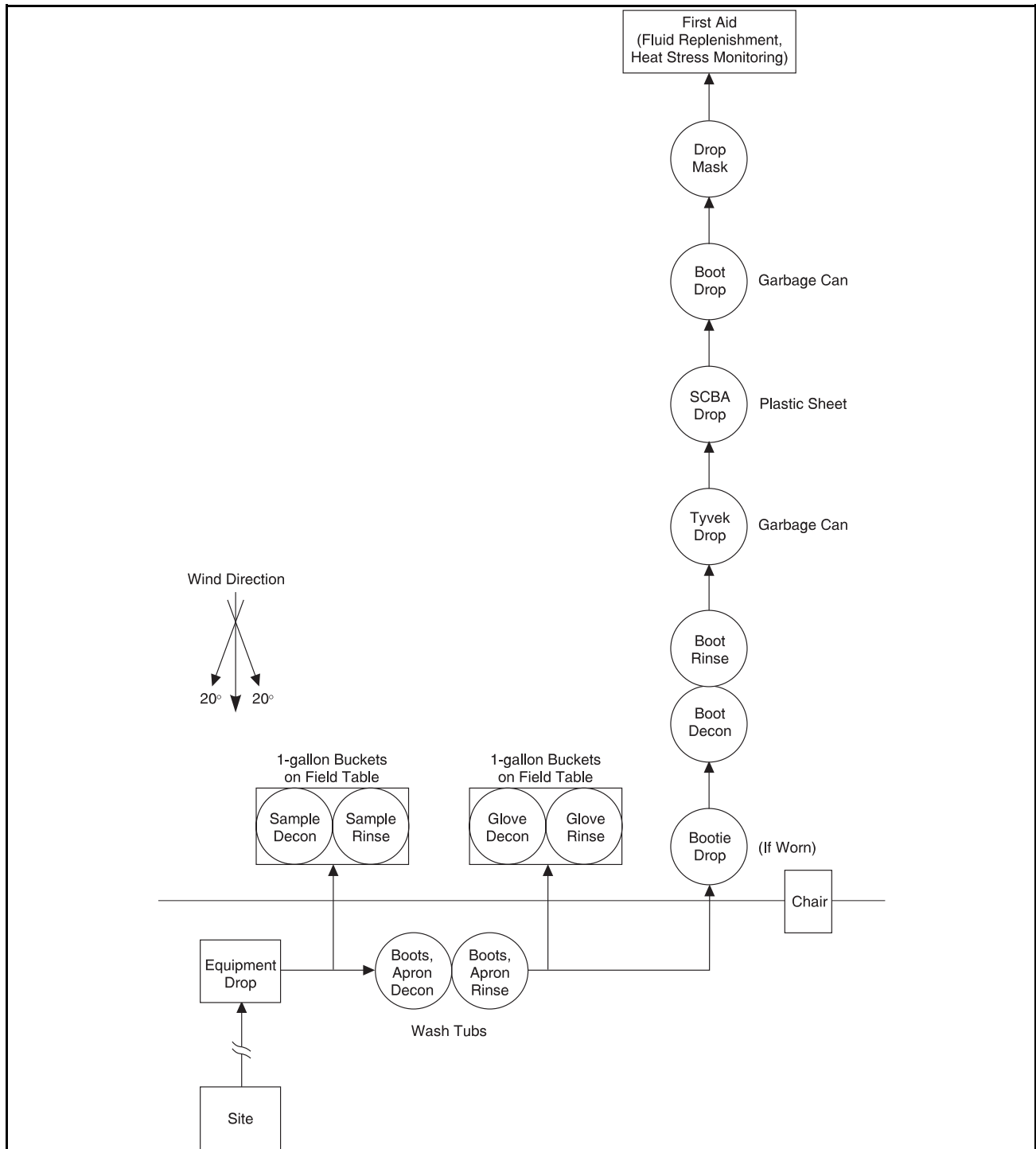
- Physical site characteristics (e.g., trees, buildings, topography);
- Associated chemical hazards;

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**Figure 4 Typical Egress from Hazardous Waste Site**

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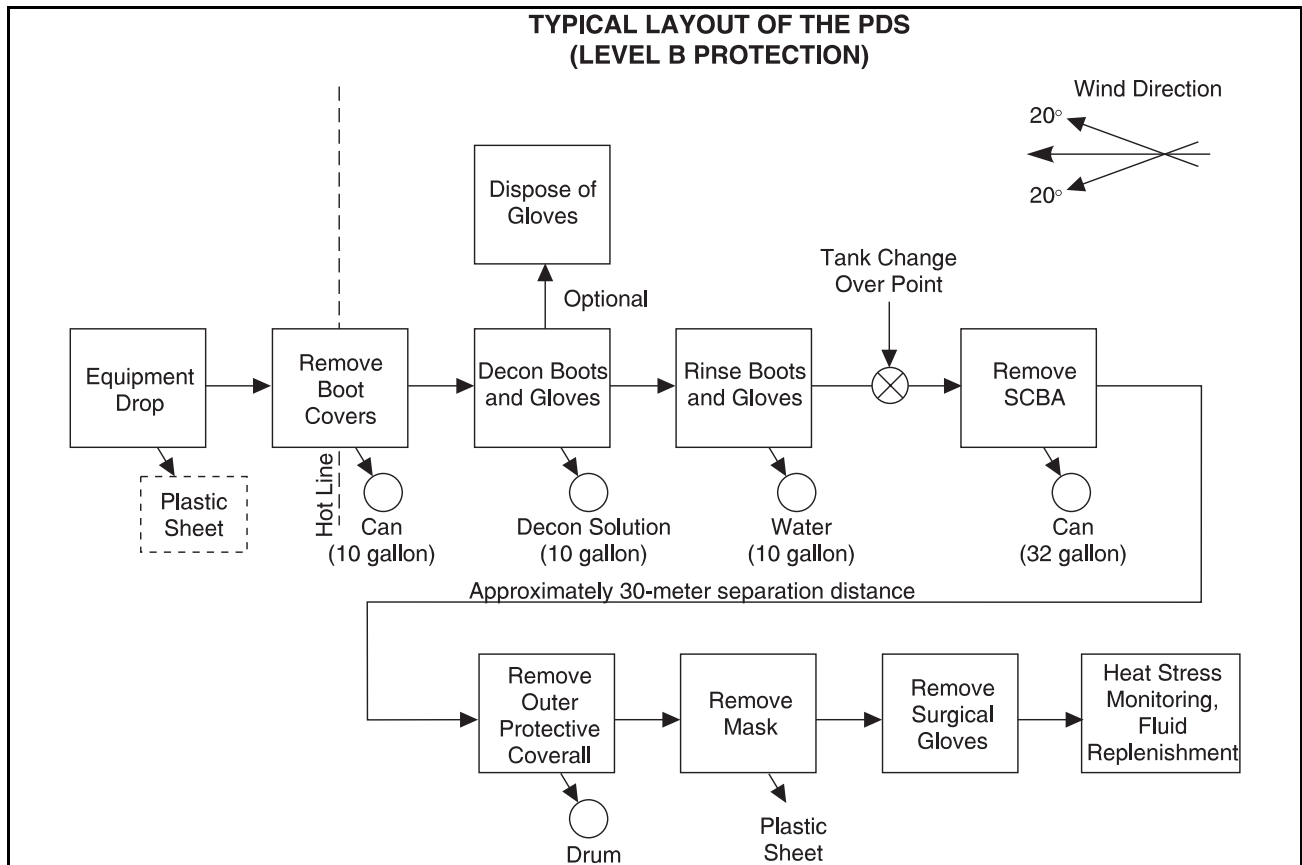


Figure 5 Typical Level B Personnel Decontamination Station

- Number of personnel entering the hot zone;
- Number of PDS personnel needed;
- Ease of PDS closure; and
- The amount of disposable materials that may be produced when closing (ground covering, cleaning materials).

3.7.4 PDS Design/Layout

The PDS should be designed so that personnel entering from the hot zone will be able to move easily from one step to the next without the possibility of missing a step and contaminating clean areas, decontamination solutions, or personnel. Signs with arrows, ropes, and/or markers delineating the proper avenue for decontaminations procedures should be visible to personnel in their working level (Level A, B, or C).



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In lieu of markers, PDS personnel may be assigned to escort and assist workers through the PDS as they exit the hot zone.

The entry point of the PDS from the hot line should be well defined and easily found by personnel while wearing their level of protection. Personnel in Level A and sometimes Level B may find it hard to read small signs and printing due to the fogging of the protective mask's face piece or the inability to turn one's head.

3.7.5 PDS Setup

The PDS itself should be set up in such a manner that will keep contamination of the ground directly underneath and adjoining it from becoming contaminated. Dropcloths or plastic sheeting will be used according to the hazards associated with the site. The sides and ends should be diked to a level that will retain the amount of decontamination fluids used in case of a spill. The boundaries of the PDS should be clearly marked using rope, tape, or flags.

Tables, chairs, and other furniture used within the PDS should be protected with plastic sheeting and decontaminated prior to removal from the site. Environmental/personal monitoring equipment should also be protected from contamination in a manner that would still afford proper usage (e.g., clear plastic bags, etc.).

3.7.6 PDS Personnel

Personnel working in the PDS, where the possibility of exposure to contaminated materials is the greatest, will be required to be dressed in a personal protective level one step lower than the personnel entering the hot zone. For example, if the working level of protection is B, then the PDS personnel working in Step 2 are required to be in Level C. Personnel working Steps 4 through 10 could be dressed in Levels C or D, depending on the associated hazard.

An adequate number of PDS personnel should be available to reduce disorganization and possible cross-contamination (e.g., persons working Step 2, outer garment decon, should not be assigned to Step 6, SCBA drop). PDS personnel should be familiar with their assigned tasks and associated hazards.

Personnel working in Steps 2 and 3 9 (hot side of the hot line) are required to complete PDS decontamination.

Under normal situations, the HSO will be stationed within the PDS and will be dressed in a personal protection level that allows him or her to enter the hot zone.

3.7.7 PDS Use

The PDS is divided into 10 steps through which personnel leaving the hot zone must utilize (see Figure 4). Depending on the associated hazards, the following steps to the basic PDS layout may be deleted or expanded as necessary.

- **Step 1: Equipment Drop.** The equipment drop is situated on the hot side of the hot line and consists basically of two plastic dropcloths; one

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labeled “C” (for clean) is for receiving and distributing clean equipment/sample materials. The other, labeled “D” (for dirty), is for receiving contaminated equipment/samples. The dirty dropcloth should be diked in a manner that will contain runoff from contaminated equipment/materials. Personnel leaving or entering the hot zone will drop off all equipment/samples used within the hot zone at this point. Contaminated equipment/samples should not be brought through the PDS.

This area may also be used for air tank changes when required. By using this area, contaminated personnel need not go through decontamination prior to tank changes if they are to immediately re-enter the area.

In the event that personnel are taking a break and will return to the contaminated area in a short period of time, certain steps (1 through 6), depending on the setup of the PDS, must first be utilized. A break area for these workers may be set aside within the PDS that will allow an avenue for re-entry.

Personnel operating the equipment drop area of the PDS should approach and stay within the contamination reduction area and should not cross the hot line.

- **Step 2: Outer Garment Decon Area.** At this step, decontamination of the outer garments is accomplished. This section contains at least two washes and one rinse. Personnel will move from the first wash area, at which time the large contaminated materials such as dirt, mud, etc., are removed and/or scraped off, to the second wash area, where large brushes, sprayers and decontamination fluids are used for further decontamination. After the second washing, a final water rinse is administration. At all times during this step of decontamination, care should be taken to avoid spilling/splashing or cross-contaminating the decontamination personnel. Fluids from this area should be kept to a minimum and collected and drummed for disposal.
- **Step 3: Bootie Removal Area.** At this step, the outer booties worn by contaminated personnel are removed. A chair or suitable item is situated on the hot side of the hot line. After the booties are removed, contaminated personnel will swivel their feet into the PDS. It should be stressed that at this point, cross-contamination from the feet of the personnel is highly likely unless special care is taken. Disposable booties should be double-bagged for disposal.



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- **Step 4: Glove and Boot Decontamination Area.** At this step, non-disposable boots and gloves are decontaminated. A single wash and rinse are normally required, but additional washes/rinses may be added depending on the amount of contamination. Personnel should be stationed here to offer assistance.
- **Step 5: Outer Garment Removal.** At this step, decontaminated outer garments are removed, and depending on the situation, either stored for future use or disposed of. Outer garments consist of hoods, full protective suits, outer boots and gloves, aprons, etc.
- **Step 6: SCBA Drop.** SCBA equipment is collected at this step. Personnel should be stationed here to assist workers in removing SCBA. Harnesses, tanks, and masks should be considered contaminated and require decontamination prior to re-use or storage.
- **Step 7: Disposable Garment Removal.** Disposable garments (Tyvek/Saranex) worn under outer garments are removed and disposed of at this step. Disposable garments should be rendered useless by cutting or tearing and double-bagged for proper disposal.
- **Step 8: Hand Wash.** All personnel will be required to wash their hands prior to exiting the PDS. One wash tub/pail and one rinse tub/pail should be sufficient. Disposable means of hand-drying is recommended (e.g., paper towels), which will be collected and double-bagged.
- **Step 9: Mask Wash.** At this step, protective masks are disassembled and washed/rinsed. It is important that the proper tools required for this task are available. To reduce the possibility of cross-contamination, protective masks are air-dried. Preventive maintenance on protective masks is also performed at this station. Replacement parts for the type and style of masks used will be available (i.e., head straps, valves, nose pieces, rubber gaskets).
- **Step 10: Field Shower.** Depending on the situation and chemical contaminant, a field shower may be required. Fluids from the shower system will be collected for future testing and disposal. Disposable means of drying will be available and collected.

3.7.8 Emergency Gross Decontamination

In cases of saving life or limb or emergency rescues, the PDS will also be utilized for performing emergency gross decontamination. At this time, routine operations of the PDS will



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cease (i.e., equipment and sample decontamination), and the focus will be on the emergency situation.

Emergency gross decontamination is defined as the immediate removal of large amounts of contamination, or removal of outer layers of protection, to a level that should not contaminate the injured individual or personnel performing emergency rescue.

Personnel performing emergency gross decontamination will be dressed in a personal protection level that is one level lower than the working party. These are normally the personnel working insteps 2 and 3, outer garment decontamination and bootie removal, because they would already be in the proper protection level.

3.7.9 PDS Closure

It is of utmost importance that the closure of the PDS be accomplished with an animal amount of contact with the PDS personnel. Possible upgrading of the contact protection (Saranex, Neoprene gloves) and respiratory protection (addition of HEPA filters) should be considered depending on the individual hazards.

All equipment, furniture, and instrumentation used within the PDS must first be decontaminated and moved to the clean area. All expendable items and decontamination solutions should be double-bagged and/or drummed according to the hazards associated and specifications in the work plan. All reusable protective clothing should be decontaminated and moved to the clean area. The PDS area is then inspected for any residual loose debris, fluids, or disposable items.

Once all items within the PDS are decontaminated and moved to the clean area, the protective ground coverings should be rolled or folded (starting from the hot line), and properly double-bagged for disposal.

Finally, PDS personnel will remove their disposable clothing starting with the coveralls, then the booties, respiratory protection, and finally, their gloves. These items will also be double-bagged for disposal.

3.8 Decontamination Solutions

Proper decontamination solutions must be used to ensure that all contamination has been removed from personnel and instruments or from other equipment that has been used on site. Some suggested decontamination solutions are shown in Table 3. The initial background study and site entry survey should provide information on proper solutions to protect against on-site chemicals. Specific decontamination chemicals (solvents) must be monitored to ensure proper respiratory protection for the PDS operator. Proper disposal of decontamination solutions must be predetermined prior to site work.

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Table 3		
PREFERRED DECONTAMINATION SOLUTIONS		
Type of Hazard	Preferred Decontamination Solution	Directions for Preparation
Inorganic acids, metal processing wastes	A	To 10 gallons of water, add 4 oz. of sodium carbonate (soda lime) and 4 pounds of trisodium phosphate. Stir until evenly mixed.
Heavy metals - mercury, lead, cadmium, etc.	A	To 10 gallons of water, add 4 oz. of sodium carbonate (soda lime) and 4 pounds of trisodium phosphate. Stir until evenly mixed.
Pesticides, fungicides, chlorinated phenols, dioxins, PCPs	B	To 10 gallons of water, add 8 oz. of calcium hypochlorite. Stir with wooden or plastic stirrer until evenly mixed.
Cyanides and other nonacidic inorganic wastes	B	To 10 gallons of water, add 8 oz. of calcium hypochlorite. Stir with wooden or plastic stirrer until evenly mixed.
Solvents and organic compounds such as trichloroethylene, chloroform, toluene, and ammonia	C (or A)	To 10 gallons of water, add 4 oz. of trisodium phosphate. Stir until evenly mixed.
PBBs and PCBs	C (or A)	To 10 gallons of water, add 4 oz. of trisodium phosphate. Stir until evenly mixed.
Oily, greasy unspecified wastes	C	To 10 gallons of water, add 4 oz. of trisodium phosphate. Stir until evenly mixed.
Inorganic bases, alkali and caustic waste	D	To 10 gallons of water, add ½ pint of concentrated hydrochloric acid. Stir with a wooden or plastic stirrer. Use eye/dermal protection.

Key:A = A solution containing 5% sodium carbonate (Na_2CO_3) and 5% trisodium phosphate (Na_3PO_4).B = A solution containing 10% calcium hypochlorite (CaCl_2O_2).C = A solution containing 5% trisodium phosphate (Na_3PO_4). This solution can also be used as a general-purpose rinse.

D = A dilute solution of hydrochloric acid (HCl).



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3.9 Communications

3.9.1 General Radio Communications Guidance

On-site communications are important in any field investigation, both for general operations and for emergency situations. Communications between a team and the CP must be maintained through the use of portable two-way radios (either hand-held or headset types). Throat microphones are available for use with respiratory protective equipment. Hand signals should be established as a contingency if the radio system fails or is unavailable.

3.9.2 Other Communications

An audible evacuation signal should also be established at the site. An air horn or automobile horn is often used for this purpose. Site-specific evacuation signals are noted in the SSP. In addition, each team member should closely adhere to the following guidelines:

- Each member should have his or her name written on the hard hat, coverall, and respirator for ease of identification when respiratory protective equipment is worn;
- Standard procedures and nomenclature will be used:
 - Each transmission is initiated by identifying the party being reached, followed by identification of the initiator, followed by a pause for acknowledgment (e.g., "Command post, this is _____, over.");
 - Each word must be pronounced clearly and language should be plain and direct. The phonetic alphabet (e.g., alpha, bravo, charlie) should be used to avoid miscommunication;
 - Each message should end with "over;" and
 - The term "out" indicates that the complete transmission is ended and no return transmission is expected.

3.10 Medical Surveillance/Monitoring

3.10.1 General Radio Communications Guidance

All E & E personnel are required to participate in the E & E corporate H&S program for toxic and hazardous substances. Medical evaluation is required:

- As soon as possible after exhibiting signs or symptoms of overexposure;



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- Upon termination of employment;
- As soon as possible following an injury; and
- As indicated by corporate Health & Safety Director.

3.10.2 Medical Monitoring During Hazardous Waste Site Operations

If circumstances require that personnel work under adverse conditions for extended periods of time, the SSO shall implement medical monitoring procedures. Such conditions (extreme heat, extreme cold, and use of chemical protective clothing) may cause subtle, though potentially dangerous, physical and mental changes that affect the safety of operations. Therefore, specific and routine monitoring of vital functions may be required.

The SSO may need to request assistance from emergency or other qualified personnel. (In most cases, this role can be filled by emergency medical personnel who have an understanding of and training in medical monitoring functions.)



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STANDARD OPERATING PROCEDURE

SAMPLING EQUIPMENT DECONTAMINATION

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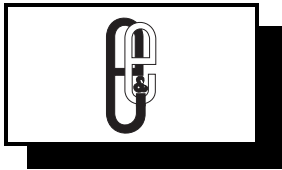
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1. Scope and Application

The purpose of this procedure is to provide a description of methods for preventing or reducing cross-contamination and general guidelines for designing and selecting decontamination procedures for use at potential hazardous waste sites. The decontamination procedures chosen will prevent introduction and cross-contamination of suspected contaminants in environmental samples, and will protect the health and safety of site personnel.

2. Method Summary

Removing or neutralizing contaminants that have accumulated on personnel and equipment ensures protection of personnel from permeating substances, reduces/eliminates transfer of contaminants to clean areas, prevents the mixing of incompatible substances, and minimizes the likelihood of sample contamination.

Cross-contamination can be removed by physical decontamination procedures. The abrasive and non-abrasive methods include the use of brushes, high pressure water, air and wet blasting, and high pressure Freon cleaning. These methods should be followed by a wash/rinse process using appropriate cleaning solutions. A general protocol for cleaning with solutions is as follows:

1. Physical removal.
2. Non-phosphate detergent plus tap water.
3. Tap water.
4. 10% nitric acid.
5. Distilled/deionized water rinse.
6. Solvent rinse.
7. Total air dry.
8. Triple rinse with distilled/deionized water.

This procedure can be expanded to include additional or alternate solvent rinses that will remove specified target compounds if required by site-specific work plans (WP) or as directed by a particular client.



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3. Interferences

The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte-free distilled/deionized water. Distilled water available from local grocery stores and pharmacies is generally not acceptable for final decontamination rinses. Contaminant-free deionized water is available from commercial vendors and may be shipped directly to the site or your hotel.

The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal water treatment system.

4. Equipment/Apparatus

The following are standard materials and equipment used as a part of the decontamination process:

- Appropriate protective clothing;
- Air purifying respirator (APR);
- Field log book;
- Non-phosphate detergent;
- Selected high purity, contaminant-free solvents;
- Long-handled brushes;
- Drop cloths (plastic sheeting);
- Trash containers;
- Paper towels;
- Galvanized tubs or equivalent (e.g., baby pools);
- Tap water;
- Contaminant-free distilled/deionized water;
- Metal/plastic container for storage and disposal of contaminated wash solutions;
- Pressurized sprayers, H₂O;
- Pressurized sprayers, solvents;
- Trash bags;
- Aluminum foil;
- Sample containers;
- Safety glasses or splash shield; and
- Emergency eyewash bottle.



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5. Reagents

There are no reagents used in this procedure aside from decontamination solutions used for the equipment. The type of decontamination solution to be used shall depend upon the type and degree of contamination present and as specified in the project/site-specific Quality Assurance Project Plan (QAPP).

In general, the following solvents are utilized for decontamination purposes:

- 10% nitric acid wash (reagent grade nitric acid diluted with deionized/distilled water – 1 part acid to 10 parts water)^a;
- Acetone (pesticide grade)^b ;
- Hexane (pesticide grade)^b;
- Methanol; and
- Methylene chloride^b.

^aOnly if sample is to be analyzed for trace metals.

^bOnly if sample is to be analyzed for organics requiring specific or specialized decontamination procedures. These solvents must be kept away from samples in order to avoid contamination by decon solvents.

6. Procedures

Decontamination is the process of removing or neutralizing contaminants that have accumulated on both personnel and equipment. Specific procedures in each case are designed accordingly and may be identified in either the Health and Safety Plan (HSP), WP, QAPP, or all three.

As part of the HSP, a personnel decontamination plan should be developed and set up before any personnel or equipment enters the areas of potential contamination. Decontamination procedures for equipment will be specified in the WP and the associated QAPP. These plans should include:

- Number and layout of decontamination stations;
- Decontamination equipment needed (see Section 4);
- Appropriate decontamination methods;
- Procedures to prevent contamination of clean areas;
- Methods and procedures to minimize worker contact with contaminants during removal of protective clothing;



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- Methods and procedures to prevent cross-contamination of samples and maintain sample integrity and sample custody; and
- Methods for disposal of contaminated clothing, equipment, and solutions.

Revisions to these plans may be necessary for health and safety when the types of protective clothing, site conditions, or on-site hazards are reassessed based on new information.

Prevention of Contamination

Several procedures can be established to minimize contact with waste and the potential for contamination. For example:

- Employing work practices that minimize contact with hazardous substances (e.g., avoid areas of obvious contamination, avoid touching potentially hazardous substances);
- Use of remote sampling, handling, and container-opening techniques;
- Covering monitoring and sampling equipment with plastic or other protective material;
- Use of disposable outer garments and disposable sampling equipment with proper containment of these disposable items;
- Use of disposable towels to clean the outer surfaces of sample bottles before and after sample collection; and
- Encasing the source of contaminants with plastic sheeting or overpacks.

Proper procedures for dressing prior to entrance into contaminated areas will minimize the potential for contaminants to bypass the protective clothing. Generally, all fasteners (zippers, buttons, snaps, etc.) should be used, gloves and boots tucked under or over sleeves and pant legs, and all junctures taped (see the Health and Safety Plan for these procedures).

Decontamination Methods

All personnel, samples, and equipment leaving the contaminated area of a site must be decontaminated to remove any chemicals or infectious organisms that may have adhered to them. Various decontamination methods will either physically remove, inactivate by chemical detoxification/disinfection/sterilization, or remove contaminants by both physical and chemical means.

In many cases, gross contamination can be removed by physical means. The physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods.



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6.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The following reviews the available abrasive methods.

Mechanical

Mechanical methods include using brushes with metal, nylon, or natural bristles. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushing, and degree of brush contact. Material may also be removed by using appropriate tools to scrape, pry, or otherwise remove adhered materials.

Air Blasting

Air blasting equipment uses compressed air to force abrasive material through a nozzle at high velocities. The distance between nozzle and surface cleaned, air pressure, and time of air blasting dictate cleaning efficiency. The method's disadvantages are its inability to control the exact amount of material removed and its large amount of waste generated.

Wet Blasting

Wet blast cleaning involves the use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using very fine abrasives, the amount of materials removed can be carefully controlled.

6.2 Non-abrasive Cleaning Methods

Non-abrasive cleaning methods work by either dissolution or by forcing the contaminant off a surface with pressure. In general, less of the equipment surface is removed using non-abrasive methods.

High-Pressure Water

This method consists of a high-pressure pump, an operator controlled directional nozzle, and high-pressure hose. Operating pressure usually ranges from 340 to 680 psi, which relates to flow rates of 20 to 140 lpm.

Steam Cleaning

This method uses water delivered at high pressure and high temperature in order to remove accumulated solids and/or oils.

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Ultra-High-Pressure Water

This system produces a water jet from 1,000 to 4,000 atm. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 m/sec. (1,000 atm) to 900 m/sec. (4,000 atm). Additives can be used to enhance the cleaning action, if approved by the QAPP for the project.

High-Pressure Freon Cleaning

Freon cleaning is a very effective method for cleaning cloth, rubber, plastic, and external/internal metal surfaces. Freon 113 (trichlorotrifluoroethane) is dense, chemically stable, relatively non-toxic, and leaves no residue. The vapor is easily removed from the air by activated charcoal. A high pressure (1,000 atm) jet of liquid Freon 113 is directed onto the surface to be cleaned. The Freon can be collected in a sump, filtered, and reused.

Physical removal of gross contamination should be followed by a wash/rinse process using cleaning solutions. One or more of the following methods utilize cleaning solutions.

Dissolving

Removal of surface contaminants can be accomplished by chemically dissolving them, although the solvent must be compatible with the equipment and protective clothing. Organic solvents include alcohols, ethers, ketones, aromatics, straight-chain alkanes, and common petroleum products. Halogenated solvents are generally incompatible with protective clothing and are toxic. Table 1 provides a general guide to the solubility of contaminant categories in four types of solvents.

Surfactants

Surfactants reduce adhesion forces between contaminants and the surface being cleaned and prevents reposition of the contaminants. Non-phosphate detergents dissolved in tap water is an acceptable surfactant solution.

Rinsing

Contaminants are removed and rinsing through dilution, physical attraction, and solubilization.

Disinfection/Sterilization

Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment and personal protective clothing.

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6.3 Field Sampling Equipment Cleaning Procedures

The following steps for equipment cleaning should be followed for general field sampling activities.

1. Physical removal (abrasive or non-abrasive methods).
2. Scrub with non-phosphate detergent plus tap water.
3. Tap water rinse.
4. 10% nitric acid (required during sampling for inorganics only).
5. Distilled/deionized water rinse.
6. Solvent rinse (required during sampling for organics only).
7. Total air dry (required during sampling for organics only).
8. Triple rinse with distilled/deionized water.

Table 1 lists solvent rinses which may be required for elimination of particular chemicals. After each solvent rinse, the equipment should be air-dried and triple-rinsed with distilled/deionized water.

Table 1	
DECONTAMINATION SOLVENTS	
Solvent	Soluble Contaminants
Water	Low-chain compounds Salts Some organic acids and other polar compounds
Dilute Bases For example: – detergent – soap	Acidic compounds Phenol Thiols Some nitro and sulfonic compounds
Organic Solvents: For example: – alcohols (methanol) – ethers – ketones – aromatics – straight-chain alkanes (e.g., hexane) – common petroleum products (e.g., fuel oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)
WARNING: Some organic solvents can permeate and/or degrade the protective clothing.	

Solvent rinses are not necessarily required when organics are not a contaminant of concern. Similarly, an acid rinse is not necessarily required if analysis does not include inorganics.

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NOTE: Reference the appropriate analytical procedure for specific decontamination solutions required for adequate removal of the contaminants of concern.

Sampling equipment that requires the use of plastic or teflon tubing should be disassembled, cleaned, and the tubing replaced with clean tubing, if necessary, before commencement of sampling or between sampling locations.

7. Quality Assurance/Quality Control

QA/QC samples are intended to provide information concerning possible cross-contamination during collection, handling, preparation, and packing of samples from field locations for subsequent review and interpretation. A field blank (rinsate blank) provides an additional check on possible sources of contamination from ambient air and from sampling instruments used to collect and transfer samples into sample containers.

A field blank (rinsate blank) consists of a sample of analyte-free water passed through/over a precleaned/decontaminated sampling device and placed in a clean area to attempt to simulate a worst-case condition regarding ambient air contributions to sample contamination.

Field blanks should be collected at a rate of one per day per sample matrix even if samples are not shipped that day. The field blanks should return to the lab with the trip blanks originally sent to the field and be packed with their associated matrix.

The field blank places a mechanism of control on equipment decontamination, sample handling, storage, and shipment procedures. It is also indicative of ambient conditions and/or equipment conditions that may affect the quality of the samples.

Holding times for field blanks analyzed by CLP methods begin when the blank is received in the laboratory (as documented on the chain of parameters and associated analytical methods).

Holding times for samples and blanks analyzed by SW-846 or the 600 and 500 series begins at the time of sample collection.

8. Health and Safety

Decontamination can pose hazards under certain circumstances even though performed to protect health and safety. Hazardous substances may be incompatible with decontamination methods (i.e., the method may react with contaminants to produce heat, explosion, or toxic products). Decontamination methods may be incompatible with clothing or equipment (e.g., some solvents can permeate and/or degrade protective clothing). Also, a direct health hazard to workers can be posed from chemical decontamination solutions that may be hazardous if inhaled or may be flammable.



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The decontamination solutions must be determined to be compatible before use. Any method that permeates, degrades, or damages personal protective equipment should not be used. If decontamination methods do pose a direct health hazard, measures should be taken to protect personnel or modified to eliminate the hazard.

All site-specific safety procedures should be followed for the cleaning operation. At a minimum, the following precautions should be taken:

1. Safety glasses with splash shields or goggles, neoprene gloves, and laboratory apron should be worn.
2. All solvent rinsing operations should be conducted under a fume hood or in open air.
3. No eating, smoking, drinking, chewing, or any hand-to-mouth contact is permitted.

9. References

Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, 1988.

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001.

Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, USEPA Region IV, April 1, 1986.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October 1985.

APPENDIX B
SUPPLEMENTAL FORMS

SAMPLE PLAN ALTERATION FORM

Project Name and Number: _____

Material to be Sampled: _____

Measurement Parameters: _____

Standard Procedure for Field Collection & Laboratory Analysis (cite references): _____

Reason for Change in Field Procedure of Analytical Variation: _____

Variation from Field or Analytical Procedure: _____

Special Equipment, Materials, or Personnel Required: _____

CONTACT	APPROVED SIGNATURE	DATE
Initiator:		
START PL:		
EPA TM:		
EPA QA Officer:		

APPENDIX C
SAMPLE DOCUMENTATION AND CHAIN-OF-CUSTODY FORMS



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International Specialists in the Environment

CHAIN-OF-CUSTODY RECORD

Page ____ of ____

[illegible]

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

*See CONCENTRATION RANGE on back of form.

234055

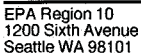


CUSTODY SEAL

Date:

Signature:





EPA Region 10
1200 Sixth Avenue
Seattle WA 98101

EPA Region 10
1200 Sixth Avenue
Seattle WA 98101

Case No.: _____

❑ Enforcement/Custody

Miscellaneous: _____

Sampling Crew: _____

Project Code: _____ Account: _____

☐ Data Confidential

[illegible]

☐ Possible Toxic/Hazardous

Proj. Off.: _____ Tel.# _____

❑ Data for STORET

Recorder:

(Signatures Required)

[illegible][illegible]



United States Environmental Protection Agency
Contract Laboratory Program

Inorganic Traffic Report & Chain of Custody Record (For Inorganic CLP Analysis)

Case No.

1. Project Code		Account Code		2. Region No.		Sampling Co		4. Date Shipped		Carrier		6. Matrix (Enter in Column A) 1. Surface Water 2. Ground Water 3. Leachate 4. Field QC 5. Soil/Sediment 6. Oil (High only) 7. Waste (High only) 8. Other (specify in Column A)		7. Preservative (Enter in Column D) 1. HCl 2. HNO3 3. NaOH 4. H2SO4 5. K2CR2O7 6. Ice only 7. Other (specify in Column D) N. Not preserved				
Regional Information				Sampler (Name)				Airbill Number										
Non-Superfund Program				Sampler Signature				5. Ship To										
Site Name				3. Purpose*				ATTN:										
City, State		Site Spill ID		Lead		Early Action		Long-Term Action										
				<input type="checkbox"/> SF <input type="checkbox"/> PRP <input type="checkbox"/> ST <input type="checkbox"/> FED		<input type="checkbox"/> CLEM <input type="checkbox"/> PA <input type="checkbox"/> REM <input type="checkbox"/> RI <input type="checkbox"/> SI <input type="checkbox"/> ESI		<input type="checkbox"/> FS <input type="checkbox"/> RD <input type="checkbox"/> RA <input type="checkbox"/> O&M <input type="checkbox"/> NPLD										
CLP Sample Numbers (from labels)		A Matrix (from Box 6) Other:	B Conc.: Low Med High	C Sample Type: Comp./ Grab	D Preservative (from Box 7) Other:	E - RAS Analysis						F Regional Specific Tracking Number or Tag Numbers		G Station Location Identifier	H Mo/Day/ Year/Time Sample Collection	I Corresponding CLP Organic Sample No.	J Sampler Initials	K Field QC Qualifier
						Diss. Metals	Total Metals	Cyanide	NO2/NO3	Low only	High only	Fluoride	pH	Conduct.				

MJY106 - TOTAL METALS

MJY107 - TOTAL METALS

MJY108 - TOTAL METALS

MJY106 - TOTAL METALS

MJY107 - TOTAL METALS

MJY108 - TOTAL METALS

MJY106 - CYANIDE

MJY107 - CYANIDE

MJY108 - CYANIDE

MJY106 - CYANIDE

MJY107 - CYANIDE

MJY108 - CYANIDE

MJY106

MJY107

MJY108

MJY106

MJY107

MJY108

MJY106

MJY107

MJY108

JN996 - EXTRACTABLE

JN997 - EXTRACTABLE

JN998 - EXTRACTABLE

JN996 - EXTRACTABLE

JN997 - EXTRACTABLE

JN998 - EXTRACTABLE

JN996 - EXTRACTABLE

JN997 - EXTRACTABLE

JN998 - EXTRACTABLE

JN996 - EXTRACTABLE

JN997 - EXTRACTABLE

JN998 - EXTRACTABLE

JN996 - VOA

JN997 - VOA

JN998 - VOA

JN996 - VOA

JN997 - VOA

JN998 - VOA

JN996

JN997

JN998

JN996

JN997

JN998

JN996

JN997

JN998

JN996

JN997

JN998

Project Name: _____ Project Code: _____ Account Code: _____

Matrix Codes (circle one only)

- 10 Water - Total
11 Water - Dissolved
17 Liquid for TCLP
40 Sediment/Soil
45 Semi-Solid/Sludge
47 Sediment for TCLP
70 Tissue
80 Oil/Solvent
00 Other - see reverse

Sample Numbers

[illegible]

Enter Matrix Code from above if other than the one circled > > > > > > > > > > > > > > >

Check the Work Group (see reverse for additional WG's) and/or specific metals below for each sample (please be sure metals checked coincide with those identified in the sampling and analysis plan).

Analy/Comp
Init/Date

Workgroups (WG):

WG 19	CLP In. Anal.
WG 32	Metals - DW-RCRA

[illegible]

Specific Metals:

Aluminum	Al
Antimony	Sb
Arsenic	As
Barium	Ba
Beryllium	Be
Cadmium	Cd
Calcium	Ca
Chromium	Cr
Cobalt	Co
Copper	Cu
Iron	Fe
Lead	Pb
Magnesium	Mg
Manganese	Mn
Mercury	Hg
Molybdenum	Mo
Nickel	Ni
Potassium	K
Selenium	Se
Silver	Ag
Sodium	Na
Thallium	Tl
Vanadium	V
Zinc	Zn
*Hexavalent Chromium	Cr+6

***The Laboratory requires advance notice prior to receiving samples for Cr+6**

Save samples after analysis? **NONE** **SOME**** **ALL**** (if **SOME**, circle sample numbers)

Special limits, methods and comments: _____

Requester's Signature

Date _____

****If samples should be saved, why?**

Note: Any element may be determined by AA or ICP.

Project Name: _____ Project Code: _____ Account Code: _____

10 Water - Total
11 Water - Dissolved
17 Liquid for TCLP
40 Sediment/Soil
45 Semi-Solid/Sludge
47 Sediment for TCLP
70 Tissue
80 Oil/Solvent
00 Other - see reverse

[illegible]

Check the Work Group (see reverse for a list of the more routine WG parameters). List any additional specific organics in the space provided, and then check off WG 60 for the analysis (be sure to only request organics that are identified in the sampling and analysis plan).

51	Volatile Organics	VOA
62	Base/Neutrals/Acids	BNA
53	Trihalomethanes	Trihal
54	Purgeable Halocarbons	Purg
6C	Resin Acids Fatty Acids Scan	
6E	Gujacols Catechols Phenolics Scan	

71 Pesticide/PCB's	Pest/PCB
72 Pesticides Only	Pest
74 PCB's Only	PCB
42 EDB	EDB
Herbicides (List each/circle on reverse)	
77 Organophosphate Pesticides (List each)	

60 Specific Organics (List below)
67 PolyAromHydro (HPLC) PAH
79 Chlorophenols (GC)
(additional space to list is provided below)

Analy/Comp
Init/Date[illegible]

ALL* (if **SOME**, circle sample numbers)

List any additional specific organics: _____

Special detection limits and comments: _____

Requester's Signature _____

Clarity

*If samples should be saved, why? _____



United States Environmental Protection Agency
Contract Laboratory Program

Organic Traffic Report & Chain of Custody Record

(For Organic CLP Analysis)

Case No.

1. Project Code		Account Code		2. Region No.		Sampling Co.		4. Date Shipped		Carrier		6. Matrix (Enter in Column A) 1. Surface Water 2. Ground Water 3. Leachate 4. Field QC 5. Soil/Sediment 6. Oil (High only) 7. Waste (High only) 8. Other (Specify in Column A)		7. Preservative (Enter in Column D) 1. HCl 2. HNO3 3. NaHSO4 4. H2SO4 5. Ice only 6. Other (Specify in Column D) N. Not preserved	
Regional Information				Sampler (Name)				Airbill Number							
Non-Superfund Program				Sampler Signature				5. Ship To							
Site Name				3. Purpose*				ATTN:							
City, State		Site Spill ID		Lead <input type="checkbox"/> SF <input type="checkbox"/> PRP <input type="checkbox"/> ST <input type="checkbox"/> FED				Early Action <input type="checkbox"/> CLEM <input type="checkbox"/> PA <input type="checkbox"/> REM <input type="checkbox"/> RI <input type="checkbox"/> SI <input type="checkbox"/> ESI				Long-Term Action <input type="checkbox"/> FS <input type="checkbox"/> RD <input type="checkbox"/> RA <input type="checkbox"/> O&M <input type="checkbox"/> NPLD			
CLP Sample Numbers (from labels)		A Matrix (from Box 6) Other:	B Conc.: Low Med High	C Sample Type: Comp Grab	D Preservative (from Box 7) Other:	E RAS Analysis VOA BNA Pest/PCB High only ARO/TOX			F Regional Specific Tracking Number or Tag Numbers		G Station Location Identifier	H Mo/Day/Year/Time Sample Collection	I Corresponding CLP Inorganic Sample No.	J Sampler Initials	K Field QC Qualifier B = Blank S = Spike D = Duplicate R = Rinsate PE = Perform. Eval. --- = Not a QC Sample
Shipment for Case Complete? (Y/N)		Page _____ of _____		Sample(s) to be Used for Laboratory QC				Additional Sampler Signatures				Chain of Custody Seal Number(s)			

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

DISTRIBUTION: Blue - Region Copy
White - Lab Copy for Return to Region

Pink - CLASS Copy
Yellow - Lab Copy for Return to CLASS

EPA Form 9110-2
(2/98)

SEE REVERSE FOR ADDITIONAL STANDARD INSTRUCTIONS
*SEE REVERSE FOR PURPOSE CODE DEFINITIONS

382028

Project Name: _____ **Project Code:** _____ **Account Code:** _____

10 Water - Total
11 Water - Dissolved
40 Sediment/Soil
45 Semi-Solid/Sludge
70 Tissue
80 Oil/Solvent
00 Other

Analy/Comp
Init/Date

Chloride	Cl
Sulfate	SO ₄
Nitrate	NO ₃
Nitrite	NO ₂
Bromide	Br

Date



Tag No.	153684	Sampler Name(s) (Please Print)	Preservative: Ice <input type="checkbox"/> No <input type="checkbox"/>	
			Chemical: Acid <input type="checkbox"/> Base <input type="checkbox"/> Other <input type="checkbox"/>	
Month/Day/Year/Time			Analyses	
			General Chemistry	
Project Code		Station Location / No.	BOD Anions Cations	
			Solids (TSS, TDS, SS)	
			COD, TOC, Nutrients	
			Oil and Grease	
			Inorganics	
			Metals	
			Cyanide	
			Organics	
			Volatiles	
			Semi-volatiles	
			Pesticides/PCBs	
			PAHs	
			Herbicides	
			Phenols	
			Other (Write In)	
CLP No.		Regional Tracking No.		
Remarks:				

APPENDIX D
COMMERCIAL LABORATORY STATEMENT OF WORK
(To be provided when completed)

APPENDIX E
TRIBUTYLTIN ANALYTICAL METHOD

A Method for Analysis of Butyltin Species and Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound

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ABSTRACT

A method for the determination of tetra-, tri-, di- and monobutyltin in marine sediments and in tissues from English sole was developed. The method utilized dichloromethane/tropolone extraction of the butyltins, derivatization with hexylmagnesium bromide and a silica/alumina column cleanup prior to gas chromatographic/mass spectrometric (GC/MS) analysis. A number of quality assurance measures were incorporated in the method. Mean recoveries of the surrogate spike triphenyltin from sediment and tissue reference materials ranged from 96% to 110%. The method was applied to sediments and livers of English sole collected in Puget Sound, Washington state. Total butyltin concentrations in sediments ranged from <5 to 1900 ng/g dry weight as Sn. Tributyltin concentrations of > 1000 ng/g were found in some sediments. Butyltins were also determined in livers from English sole captured at sites where sediments were contaminated with butyltins. Dibutyltin (at concentrations up to 870 ng/g dry weight as Sn) was the predominant butyltin species found in the livers.

INTRODUCTION

During the past several years, concern over the presence and impact of butyltin compounds in marine environments has escalated. Tributyltin



(TBT) can enter the marine environment not only through its use in antifouling paints, but also through its other applications which include disinfection of industrial and electrical generation plant cooling waters, slime control in paper mills, and as stabilizers of polyvinyl chloride (PVC) plastics (Snoey *et al.*, 1987). Tributyltin effectively inhibits the growth of fouling organisms that attach to boat hulls, but unfortunately also exhibits toxicity toward non-target organisms (e.g. detrimental effects in oysters have been observed at water concentrations of ~ 10 ng/liter (Walsh *et al.*, 1985, Hall & Pinkney, 1985; Thain *et al.*, 1987)).

Dibutyltin (DBT) and monobutyltin (MBT) are formed as degradation products of environmental TBT. In addition, these two compounds are of economic importance for their uses in PVC plastics and also can be introduced into the marine environment in industrial wastes. Although DBT and MBT are apparently less harmful to aquatic life than TBT (Hall & Pinkney, 1985, Walsh *et al.*, 1985), DBT has been found to be hepatotoxic and immunotoxic in rodents (Snoey *et al.*, 1987), and monobutyltin leads to steatosis and liver enlargement in mice. Moreover, tetraorganotin compounds can also produce deleterious effects similar to the corresponding triorganotins, but the effects are often delayed and less severe (Snoey *et al.*, 1987). Thus, an analytical method which can identify and quantitate all of the individual butyltin species present in a sample is needed.

The methodology for the trace determination of butyltins in water samples is well documented (Meinema *et al.*, 1978, Mathias *et al.*, 1986, Blair *et al.*, 1986a, Valkirs *et al.*, 1986, Unger *et al.*, 1986). Methods for the measurement of some of the butyltin species in sediments and tissues have also been reported (e.g. Hattori *et al.*, 1984, Maguire, 1984; Tsuda *et al.*, 1986, Muller, 1987, Rice *et al.*, 1987, Short, 1987, Cooney *et al.*, 1988, Han & Weber, 1988, Sullivan *et al.*, 1988, Stephenson & Smith, 1988). These methods have (1) employed a variety of solvents to extract the butyltins (hexane, benzene, ethyl acetate, ethyl ether, dichloromethane and methanolic hydrochloric acid), (2) included different types of derivatives to increase volatility (hydrides, ethyl-, pentyl- and hexyl-derivatives) and (3) used a number of analytical techniques to quantitate butyltins (graphite furnace/atomic absorption (GF/AA), gas chromatography/atomic absorption (GC/AA), gas chromatography flame photometry (GC/FPD), hydride generation/atomic absorption (HG/AA) and gas chromatography/mass spectrometry (GC/MS)). However, none of the reported methods measures all four butyltin species and many have not included adequate quality assurance (QA) procedures to establish the reliability of the data. The method described in this paper uses dichloromethane/tropolone to extract the four butyltin species, uses hexyl derivatives formed by Grignard reaction and, following silica/alumina column cleanup, analyzes the hexylbutyltins

by GC/MS. A high level of QA is included in the method. Using the method, butyltins were determined in sediments and livers of English sole collected in Puget Sound, Washington state.

MATERIAL AND METHODS

Reagents

Tetrabutyltin, tributyltin chloride, dibutyltin dichloride, monobutyltin trichloride, tin(IV)chloride, tropolone and hexylmagnesium bromide (2M in diethyl ether, in Aldrich Sure/Seal™ bottle) were obtained from Aldrich (Milwaukee, WI). Tripropyltin chloride and pentylmagnesium bromide (1.5M in diethyl ether) were obtained from Alfa Products (Danvers, MA). Dichloromethane, hexane and pentane were of Burdick and Jackson High Purity grade. All other reagents were of ACS Reagent grade or better. Silica (grade 923, 100–200 mesh from MCB, Norwood, OH) and alumina (Type F-20, 80–200 mesh from Sigma Chemical, St Louis, MO) were heated at 700 °C for 18 h, stored at 170 °C and, prior to use, cooled to room temperature in a desiccator. Amino Sep-Paks™ were from Waters Chromatography Division (Milipore Corp., Milford, Mass.).

Preparation of triphenyltin chloride (TPeT) surrogate spike

Surrogate spikes (or internal standards) are added to samples to measure losses that may occur during sample workup (extraction, derivatization, etc.). The surrogate spike should be chemically similar to the analytes and should be added at known concentration to the original sample prior to extraction. To prepare the TPeT surrogate spike used in our studies, 1 ml of pentylmagnesium bromide was added dropwise to a solution of ~300 mg of tin(IV)chloride in hexane. The mixture was allowed to react at room temperature for 30 min. Concentrated HCl was then added dropwise until a clear solution was obtained. The hexane phase (containing primarily tetraphenyltin) was chromatographed using a 1.5 cm by 30 cm glass column dry-packed with 10 g silica and the tetraphenyltin eluted with 25 ml hexane. The purified tetraphenyltin (~400 mg dissolved in 5 ml hexane) was then treated with ~100 mg tin(IV)chloride, added dropwise. The resulting mixture of tetra-, tri- and monopentyltin was partitioned with 10 ml distilled water. The hexane phase containing unreacted tetraphenyltin and TPeT was chromatographed on silica as before. The tetraphenyltin was eluted with 25 ml hexane and the TPeT was eluted with 25 ml dichloromethane. A

portion of the TPET was dissolved in hexane, derivatized with hexylmagnesium bromide and checked for purity by GC/MS. A solution containing 50 ng TPET/ μ l was prepared as a spiking solution.

Sediment extraction procedures

Two different extraction techniques (refluxing and tumbling) and two different solvents (hexane and dichloromethane) were tested for their abilities to extract butyltins from sediments. For extraction of the sediments using the solvent reflux procedure, wet sediment (10 g, acidified to pH 2 with HCl to remove carbonates and sulfides) and TPET (100 μ l) were added to 75 g granular anhydrous sodium sulfate (Na_2SO_4) in a mortar and mixed with a pestle to produce a dry, free-flowing powder. The powder was refluxed with 200 ml dichloromethane or hexane (each containing 0.1% tropolone) for 1 h. Tropolone is a complexing agent which aids in the extraction of the organotin chlorides. The sediment extracts were filtered through a glass Buchner funnel with a medium-porosity fritted disc using water aspiration suction. The sediment/ Na_2SO_4 mixture was washed twice with 50 ml portions of the solvent. The combined extracts were concentrated to ~5 ml by evaporation on a 50 °C water bath. The dichloromethane was exchanged to hexane (5 ml) for derivatization of the organotins.

For the extraction of sediments by tumbling, anhydrous Na_2SO_4 (50 g), 100 ml dichloromethane or hexane (each containing 0.1% tropolone), 10 g sediment (acidified to pH 2 with HCl), and 100 μ l TPET surrogate spike (and 100 μ l tripropyltin chloride (TPrT) in some samples) were added to 250 ml amber-glass round bottles with teflon-lined caps. The bottles were shaken to mix the contents and then rolled on a tumbler overnight (16 h). The solvent was decanted and filtered as above. An additional 100 ml of solvent/tropolone were added and the bottle rolled for ~6 h. Again the solvent was filtered and the sediment/ Na_2SO_4 mixture washed two times with 25 ml portions of solvent. The extracts were combined, concentrated and, if needed, exchanged to hexane as above.

Tissue extraction procedure

For the tissue extraction, English sole liver or oyster reference material (3.0 ± 0.5 g), 25 ml dichloromethane with 0.1% tropolone, 20 g anhydrous Na_2SO_4 , 100 μ l TPET and 100 μ l TPrT were added to a 100 ml glass centrifuge tube. The sample was macerated/extracted using a Tekmar Tissumizer for 0.5 min with a tissumizer setting of 80 and then for 1.5 min at a setting of 60. The solvent was decanted into a fritted glass filter funnel and filtered using suction. An additional 25 ml dichloromethane/tropolone were

added to the centrifuge tube and the extraction repeated. After decanting the second extract, the Na_2SO_4 /sample mass was washed with 10 ml dichloromethane by mixing on a Vortex Genie for 10 s. The solvent was decanted and the combined extracts concentrated and exchanged to hexane as above.

Organotin derivatization, extract cleanup and GC/MS analysis

Hexyl derivatives of the organotins in sediment and tissue extracts were formed by addition of 0.5 ml hexylmagnesium bromide Grignard reagent. The mixture was allowed to react for 30 min at room temperature under a blanket of nitrogen gas. The unreacted Grignard reagent was then destroyed and solubilized by dropwise addition of concentrated HCl until a clear solution resulted. The hexane layer containing the derivatized organotins was chromatographed on silica/alumina using a glass column (1.5 cm by 30 cm) dry-packed with 4.5 g each of alumina and silica, and the organotins eluted with 20 ml pentane. The pentane was evaporated under a stream of nitrogen gas to ~1 ml and transferred to GC vials. For tissue extracts, an additional Sep-PakTM cleanup was performed prior to GC/MS analysis as follows. The tissue extract was loaded on the amino Sep-PakTM and washed through with 3 ml pentane. The pentane eluate was concentrated to 1 ml and then transferred to a GC vial.

Hexamethylbenzene GC internal standard was added to all vials (final concentration 5 ng/ μl) and the solutions analyzed by GC/MS (Hewlett Packard 5840 GC with a bonded phase DB-5 fused silica capillary column (0.25 mm i.d.), interfaced to a Finnigan 3200 MS with an INCOS data system operated in the electron impact mode). The carrier gas was helium and the flow rate was 3 ml/min. Injection was splitless for 0.30 min and initial column temperature (50 °C) was held for 0.85 min and then increased at a rate of 8 °C/min to 300 °C. Mass spectra and GC retention time afforded identification of the alkyltins. The MS was scanned from 40–510 amu in 1 s and the ions $m/e = 203$ to 207 and $m/e = 190$ to 193 used to quantitate the surrogate spikes TPrT and TPtT, respectively. The ions $m/e = 176$ to 179 were used to quantitate tetrabutyltin, TBT and DBT, and the ions $m/e = 204$ to 207 used to quantitate MBT.

Preparation of GC/MS calibration standards

To synthesize the butylhexyltin standard materials, several hundred milligrams of each of the butyltin chlorides (in hexane) were treated separately with 1 ml hexylmagnesium bromide and allowed to react for 30 min at room temperature. Hydrochloric acid was added dropwise as

previously described and the hexane phase (containing the hexylbutyl derivatives) chromatographed on silica (using a 1.5 cm by 30 cm glass column containing 10 g silica and eluting the organotins with 25 ml hexane). The hexane was removed under a stream of nitrogen gas and weighed amounts of the derivatives were diluted with hexane and used for calibration standards.

Sediment and tissue reference materials

Two sediment reference materials (RM) and one tissue RM were previously prepared in our laboratory as part of NOAA's National Status and Trends Program (NS&T), primarily for use as an RM for aromatic hydrocarbons (AH) and chlorinated hydrocarbons (CH). A large sediment sample was collected in the Duwamish Waterway, Seattle, WA (called Du III), mixed in a cement mixer for 4 h, placed in 4 oz glass jars, and stored at -20°C (Krahn *et al.*, 1988). This sediment represents a contaminated environmental reference sediment which contains all four butyltins. The second reference sediment was prepared by addition of TBT (as well as AH and CH) to sediments from a non-urban area, Sequim Bay, Washington (called SQ). This sediment was chosen because of its lack of organotins (as well as aromatic hydrocarbons, PCBs and pesticides) and its similarities to Du III in total organic carbon and grain size.

The oyster tissue RM (called OI) was prepared by homogenizing whole oysters (*Crassostrea gigas*), adding a solution that contained analytes of interest, including TBT, and re-homogenizing (Krahn *et al.*, 1988). The homogenate (~ 10 g) was placed in 20 ml vials with teflon-lined caps and stored at -20°C .

Collection of environmental samples

Sediments were collected (using a modified Van Veen grab) from the Duwamish West Waterway (three stations, called Duwamish Waterway 1, 2, and 3), from Elliott Bay along the Seattle Waterfront (three stations, from Piers 65 to 69, called Seattle Waterfront 1, 2 and 3), and from the reference area President Point (three stations, called President Point 1, 2 and 3). The distance between the three stations at each site was about 0.25 nautical miles (Fig. 1). Surface samples of the top 2 cm of sediment were collected in glass jars, stored on ice, and frozen at -20°C upon return to the laboratory. Sediments from the nine stations were analyzed in triplicate.

The Duwamish West Waterway was chosen because it is a major urban waterway and an area of intense industrial activity which includes ship building, maintenance and repair facilities, airplane manufacturing, asphalt

Fig. 1. Location of sampling sites in Elliott Bay.

and cement. This site has been found to have high levels of PCBs and organotins (Krahn *et al.*, 1984). President Point is an anthropogenic area with a large English trawl fishery. The speed of a trawl at each site is about 1 km/h and the capture area is about 100 m².

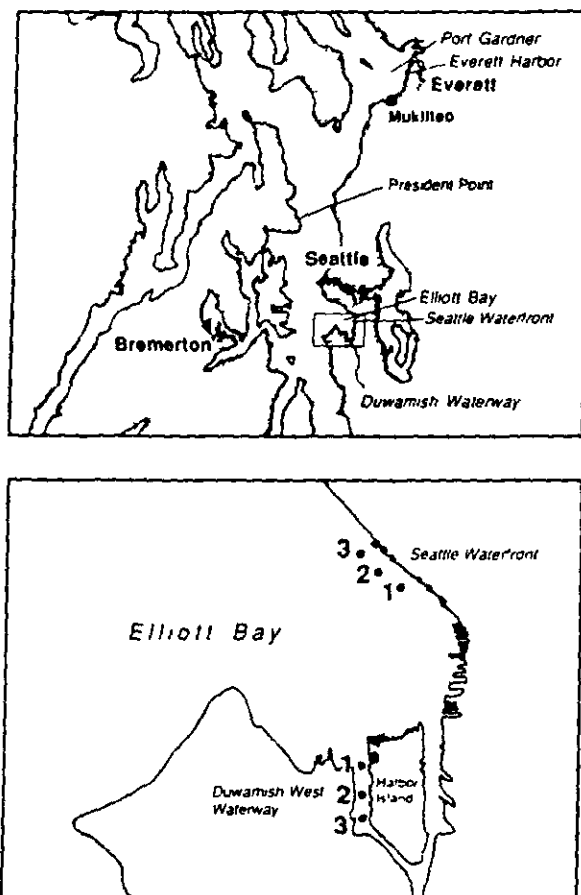


Fig. 1 Locations of sampling sites in Puget Sound. Closed circles (●) on enlarged map of Elliott Bay show stations in the Duwamish West Waterway and along the Seattle Waterfront where sediments were collected

and cement plants, scrap metal yards, oil tank farms and port facilities. This site has been found to be highly contaminated with aromatic hydrocarbons, PCBs and metals including cadmium, lead, arsenic and mercury (Malins *et al.*, 1984). The Seattle Waterfront site is near a Washington State ferry terminal and combined sewage overflow and/or storm drains. President Point is a non-urban site (reference area) with no known sources of anthropogenic chemicals.

English sole were also captured at the above three sites using an otter trawl. The individual trawls at each station were 10 min in duration at a speed of about 2.5 knots and followed a path connecting the three stations at each site. The fish were weighed, measured and necropsied within 1 h of capture and the liver tissues were frozen in liquid nitrogen. All samples were

stored at -20°C until analyzed. Livers from five fish were composited and analyzed in triplicate.

Quality assurance (QA) procedures

The QA procedures included the use of surrogate spikes, calibration standards, blanks, spiked samples, reference materials and replicate analyses to evaluate performance. Surrogate spikes (TPeT and in some cases also TPrT) were added to all samples, reagent blanks and spiked blanks. Repetitive analyses of RM were carried out to evaluate the performance of the method. Environmental samples were analyzed in triplicate and the concentrations reported based on TPeT surrogate spike.

RESULTS

Method for butyltin analysis in sediments and tissues

Table 1 shows the recovery of TPeT surrogate spike from blanks and sediment using different extraction methods and solvents. The tumbling method (using either dichloromethane or hexane) resulted in a higher recovery of TPeT compared to the reflux method. The recovery of butyltins from spiked reagent blanks was also generally greater for the tumbling procedure, although not statistically significant (Table 2). Moreover, the relative standard deviations (RSD) for the recovery of TPeT from sediments and blanks (Table 1) were usually lower for the tumbled (RSD ranged from 9 to 28%) versus the refluxed samples (RSD ranged from 30 to 63%). The

TABLE 1
Recovery of Surrogate Spike^a Using Different Extraction Techniques. Per cent Recovery of Triphenyltin \pm Standard Deviation

Sample	Tumble CH_2Cl_2	Tumble hexane	Reflux CH_2Cl_2 ^b
Reagent blank	86 ± 22 ($n = 4$)	64 ± 18 ($n = 2$)	53 ± 25 ($n = 2$) ^c
Spiked blank	89 ± 18 ($n = 9$)	88 ± 8 ($n = 3$)	75 ± 24 ($n = 5$) ^d
SQ	99 ± 20 ($n = 13$)	88 ± 15 ($n = 3$)	43 ± 27 ($n = 7$) ^e
Du III	96 ± 7.6 ($n = 7$)	not tested	27 ± 8.0 ($n = 3$) ^f

^a Added at a concentration equivalent to 500 ng/g sediment wet weight.

^b CH_2Cl_2 = dichloromethane.

^c Significantly different from tumble/dichloromethane, $p < 0.05$, (unpaired, two-tailed Student *t*-test).

^d Not significantly different from tumble/dichloromethane, $p = 0.25$.

^e Significantly different from tumble/dichloromethane, $p < 10^{-4}$.

Per cent
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Triphenyltin
Dibutyltin
Monobutyltin
Triphenyltin

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TABLE 2

Per cent Recovery of Butyltin Chlorides from Spiked Blanks^a using Tumbling or Reflux Extraction Techniques Per cent Recovery \pm Standard Deviation of the Mean

Compound	Tumble CH_2Cl_2 (n = 9)	Tumble/hexane (n = 3)	Reflux, CH_2Cl_2 ^b (n = 5)
Tetrabutyltin	95 \pm 10	86 \pm 15	80 \pm 18
Tributyltin	89 \pm 14	71 \pm 12	74 \pm 21
Dibutyltin	78 \pm 23	73 \pm 12	63 \pm 7
Monobutyltin	39 \pm 21	12 \pm 12	64 \pm 15 ^c
Triphenyltin	89 \pm 18	88 \pm 8	75 \pm 24

^a Butyltins added at concentrations corresponding to 500 ng/g

^b CH_2Cl_2 = dichloromethane

^c Significantly greater than tumble CH_2Cl_2 or tumble/hexane ($p = 0.05$)

recovery of TPET was not significantly different when added at concentrations of 300 to 800 ng TPET/g sediment. Tropolone at 0.1% w/v in the extraction solvent was found to be an effective complexing agent necessary for the extraction of organotin chlorides (e.g. without tropolone the recoveries of TBT, DBT, MBT and TPET were 20% or less for spiked blanks).

Using the tumbling procedure, hexane and dichloromethane performed equally well for the recovery of all butyltins except MBT from spiked reagent blanks (Table 2). Only for MBT was recovery < 70% when using the tumbling procedure. This suggests that the concentration of MBT determined in sediments using tumbling may be underestimated. Dichloromethane extracted a greater proportion of MBT than hexane did for the tumbled spiked blank ($p = 0.066$). It also extracted TBT from a sediment matrix more effectively than hexane did (e.g. the concentrations of TBT in the SQ RM were found to be 31 ± 12 (n = 13) and 11 ± 5.7 ng/g dry weight as Sn (n = 3) using dichloromethane and hexane, respectively, with the tumbling procedure (significantly different, $p < 0.02$). Although the reflux extraction with dichloromethane appeared to extract MBT from spiked blanks more effectively, the generally lower RSD and higher butyltin and TPET recoveries for the tumbling procedure led to its choice as the best procedure for analyzing sediments.

The procedure for analysis of butyltins in tissues (using a Tissumizer and dichloromethane tropolone) yielded high recoveries of butyltins and surrogate spikes. In oyster tissue spiked with butyltins at ~400 ng/g wet weight, the recoveries of tetrabutyltin, TBT, DBT, MBT and TPET were $74 \pm 17\%$, $80 \pm 13\%$, $93 \pm 19\%$, $72 \pm 22\%$ and $116 \pm 13\%$, respectively (n = 4).

TABLE 3
Butyltin Concentrations in Sediments from Puget Sound^a

Site, station	n	Concentration (ng/g dry weight as Sn) ^b				% TPeT recovery
		Tetraethyltin	Tributyltin	Dibutyltin	Monobutyltin	
Duwamish Waterway 1	3	14 ± 1.0	610 ± 61	220 ± 51	87 ± 49	100 ± 10
Duwamish Waterway 2	3	25 ± 1.3	1 300 ± 290	470 ± 150	150 ± 130	93 ± 3.0
Duwamish Waterway 3	3	16 ± 3.1	570 ± 83	210 ± 54	49 ± 11	91 ± 4.0
Seattle Waterfront 1	3	<0.63 ± 0.25	36 ± 3.0	28 ± 2.6	66 ± 2.4	88 ± 3.5
Seattle Waterfront 2	3	<3.9 ± 3.7 ^c	120 ± 4.0	83 ± 5.3	22 ± 8.4	79 ± 7.0
Seattle Waterfront 3	3	<9.7 ± 4.5 ^c	55 ± 8.9	57 ± 18	13 ± 9.3	84 ± 1.3
President Point 1	3	<0.36 ± 0.22	<0.85 ± 0.56	<2.6 ± 1.0	<0.8 ± 0.14	140 ± 12
President Point 2	3	<0.45 ± 0.22	<0.86 ± 0.24	<3.9 ± 2.7	<1.9 ± 1.4	99 ± 18
President Point 3	3	<0.36 ± 0.25	<0.40 ± 0.11	<3.8 ± 2.3	<0.89 ± 0.51	130 ± 4.0

^a The tumbling procedure with dichloromethane tropolone was used.

^b Each value in the table is the mean ± standard deviation for triplicate analyses.

^c Presence of interfering peaks in these samples increased detection limits.

Analyses of butyltins in sediments and fish tissues

The butyltin concentrations in sediments from three sites in Puget Sound are shown in Table 3. The highest concentrations of TBT were found in sediments from the Duwamish Waterway. The concentrations of DBT and MBT in these sediments were approximately 3 to 10 times less than the concentration of TBT, respectively. Tributyltin constituted 66% of the total butyltins present in the Duwamish Waterway sediment. Butyltins were also found in sediments from the three Seattle Waterfront stations. In these sediments, however, the concentrations of DBT (41% of total butyltins) were comparable to the TBT concentrations (49% of total butyltins). Butyltins were not detected (detection limits were all < 3.9 ng/g dry weight) in the samples from the President Point reference site.

Butyltins were present in liver tissue from English sole captured in the Duwamish West Waterway and the Seattle Waterfront sites (Table 4). The results for analysis of OLRM are also shown in Table 4. Unlike the sediment samples, where TBT was found in higher concentrations than the other butyltins, DBT was the predominant species measured in the livers. Tributyltin was detected only in the livers from fish captured at the Duwamish site and, on a wet weight basis, was about 18 times lower than the average TBT concentration in the Duwamish sediments. The additional cleanup step for tissue extracts using an amino Sep-PakTM was necessary to remove GC/MS interferences and resulted in detection limits generally less than 20 ng/g dry weight as Sn. However, the detection limit for MBT in the Duwamish English sole livers was 55 ng/g due to an unidentified

Butyl

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Tribu
Dibu
MonTPeT
TPrT

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tetrabutyl

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TABLE 4
Butyltins in Livers of English Sole Captured in Puget Sound and in Oyster Reference Material (RM) Butyltin concentration (ng/g dry wt as Sn)

Compound	Duwamish Waterway (n = 3)	Seattle Waterfront (n = 2)	President Point (n = 3)	Oyster RM (n = 4)
Tetrabutyltin	$<40 \pm 0.47$	$<12 \pm 12$	$<23 \pm 0.29$	$<46 \pm 4.9$
Tributyltin	100 ± 9.5	$<22 \pm 25$	$<84 \pm 10$	160 ± 64
Dibutyltin	870 ± 68	190 ± 42	$<32 \pm 7.4$	$<30 \pm 6.5$
Monobutyltin	$<55 \pm 28^*$	$<15 \pm 17$	$<58 \pm 4.1$	$<76 \pm 5.4$
TPeT recovery	$52\% \pm 10$	$82\% \pm 7.8$	$70\% \pm 20$	$110\% \pm 14$
TPrT recovery	$65\% \pm 14$	$76\% \pm 11$	$66\% \pm 18$	not added

* Presence of interfering peaks in the sample increased detection limits

chromatographic interferent and suggests that for certain atypical liver samples, additional cleanup may be needed if low concentrations of MBT are of interest

Overall, the QA results for the analyses of sediment and tissues were consistent. The mean recovery of TPeT for the 27 Puget Sound sediment analyses was $101 \pm 22\%$. TPrT was added to more recently analyzed samples as a second surrogate spike and its recovery was comparable to that of TPeT in sediment (e.g. $96 \pm 11\%$ in Du III ($n = 6$)) and tissues (see Table 4). The mean RSD for butyltins in the triplicate analyses of sediments were generally $<20\%$ except for MBT (mean RSD = 52%). The limits of detection (mean blank concentration plus three times the standard deviation) for the sediment procedure were 0.67, 0.49, 0.19 and 0.34 ng/g as Sn for tetrabutyltin, TBT, DBT and MBT, respectively ($n = 9$). Analysis of SQ reference material was performed with each set of sediment samples and yielded TBT concentrations generally less than one standard deviation different than results of our initial repetitive analyses of SQ (30 ± 13 ng TBT/g dry weight as Sn, $n = 10$). For tissues, the mean RSD for DBT was 15% and for TBT was 9.5% in the one sample which contained TBT. For the tissue procedure, the limits of detection (mean blank concentration plus three times the standard deviation) were 2.0, 1.4, 9.8 and 1.3 ng/g as Sn for tetrabutyltin, TBT, DBT and MBT, respectively ($n = 4$).

DISCUSSION

We report the first comprehensive method for the analysis of all four butyltin species in marine sediments and in tissues from marine organisms

The method also includes a number of QA procedures. Sediments from three sites in Puget Sound, and livers from English sole captured at the three sites were analyzed using the method. Tributyltin concentrations in the sediments ranged from <0.4 to 1300 ng/g dry weight as Sn. Tributyltin made up a greater proportion of total butyltins in the Duwamish Waterway sediment than in the Seattle Waterfront sediment. Unlike the sediment, DBT was found to be the predominant butyltin species present in English sole livers.

The use of dichloromethane (with 0.1% tropolone) with tumbling (sediment) or homogenizing (tissues) was the most effective extraction technique (e.g. using this method, the recoveries of butyltins and surrogate spikes were generally the greatest and the RSD for replicate analyses were the smallest). The use of dichloromethane has additional benefits as extraction solvent compared to other solvents that have been applied to sediments and tissues in previously reported methods. For example, tropolone dissolves more readily in dichloromethane than in hexane, dichloromethane is non-flammable and it has a relatively low boiling point.

Our method exhibits a number of other advantages. Quantitation and confirmation of butyltin species were based on GC/MS analysis of highly stable hexyl derivatives formed from the alkyltin chlorides in the samples. The hexyl derivatives of organotin are much more stable than the hydride derivatives (Mathias *et al.*, 1987) which were used in the Tsuda *et al.* (1986) and Hattori *et al.* (1984) methods for analysis of sediments and tissues. The hydrides' labile nature limits the use of cleanup steps necessary for removal of interfering compounds that are present in extracts of complex matrices (such as sediment or fish tissues (Zuckerman *et al.*, 1978)). The hydrides are very volatile and much more prone to evaporative losses during concentration than the hexyl derivatives. Methyl- and ethyl-derivatives of butyltins (as used by Muller, 1987) are also subject to significant losses during evaporation and concentration steps (Maguire & Huneault, 1981). Sodium sulfate is a rapid and effective drying agent and was used in our method rather than air-, oven- or freeze-drying. The two latter techniques are reported to lead to TBT losses of up to 60% (Rice *et al.*, 1988).

Each of the previously published methods for analysis of butyltins in sediments and tissues has some positive features. For example, some methods use dichloromethane as the extraction solvent (Sullivan *et al.*, 1988), some include tropolone as a complexing agent to improve extraction efficiency (Maguire *et al.*, 1986; Muller, 1987; Sullivan *et al.*, 1988), some use the more stable, less volatile pentyl or hexyl derivatives (Maguire *et al.*, 1986; Rice *et al.*, 1988) and some include the addition of surrogate spikes (Muller, 1987; Han & Weber, 1988; Rice *et al.*, 1987; Sullivan *et al.*, 1988). Our method incorporates all these advantages plus has the ability to measure all four butyltin species and includes the additional quality control technique of RM analysis.

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Keeping in mind that different methods were used for analysis, the concentrations of butyltins we report in sediments from Puget Sound can be compared with sediment butyltin concentrations reported by others. Maguire *et al* (1986) analyzed a total of 235 sediments from across Canada (air dried sediments were extracted with benzene/tropolone and pentyl derivatives were formed) Tributyltin was detected in 78 of the sediments, primarily those collected in harbors and lakes where boating and shipping traffic were heavy Dibutyltin and MBT were found in 61 and 47 of the 235 sediments, respectively The concentrations and proportions of butyltins in sediments from the Duwamish West Waterway are consistent with those reported by Maguire *et al* (1986) in sediments from Vancouver Harbor As with our findings for Puget Sound sediments, TBT was generally the predominant species of butyltin found in the Vancouver Harbor sediments (at levels up to 10 800 ng/g as Sn, dry weight), with DBT and MBT reported less often In contrast to these results, freshwater lake and river sediments which were collected by Maguire *et al* (1986), contained MBT as the predominant butyltin, with DBT often present in greater amounts than TBT Tsuda *et al* (1986) used the hydride derivatives to detect TBT and DBT in sediments from Lake Bawa in Japan As with the freshwater sediments analyzed by Maguire *et al* (1986), Tsuda *et al* (1986) also reported higher DBT than TBT levels in lake sediments

Among the factors which can affect the relative proportions of butyltins in sediments are the rates and types of degradation reactions (which may differ in saltwater and freshwater environments), the butyltin profile of sources, and processes such as volatilization, resuspension, adsorption and desorption Certain butyltin paint formulations contain significant amounts of DBT and tetrabutyltin that can leach into marine waters (Blair *et al*, 1986b) and eventually be adsorbed onto sediments Degradation of alkyltins proceeds generally by successive dealkylation to form inorganic tin as the final product (Maguire & Tkacz, 1985) Microbiologically mediated degradation of TBT and sedimentation appear to be the major processes involved in the disappearance of TBT from the water column (Maguire *et al*, 1983, Hinga *et al* 1987) Algae, bacteria and fungi are all capable of degrading tributyltin in water (Lee *et al*, 1987, Barug, 1981, Olson & Brinckman, 1986) Some organisms produce DBT, while others produce MBT as the major degradation product of TBT The presence or absence of TBT-metabolizing microalgae and bacteria may lead to marked differences in the environmental persistence and profile of butyltins in different waters and sediments

The heterogeneous nature of the distribution of butyltins in the environment is evidenced by the results at both the Duwamish and Seattle Waterfront sites In each case, one of the stations showed significantly higher TBT concentrations than the other two (e.g. the level of TBT at station No 2

in the Duwamish was two times that of the other stations) Butyltin concentrations in tissues of territorial benthic fishes may be more representative of the mean butyltin levels in an area. Hence it could be important to measure butyltin concentrations in benthic organisms such as fish, as well as in sediment, to obtain a more complete picture of environmental contamination and its consequences.

Dibutyltin was the predominant butyltin species detected in livers from English sole captured in the Duwamish Waterway. There have been no other reports of butyltin levels in livers of fish captured in the field. Davies & McKie (1987) did report that total tin levels were increased in the livers of Atlantic salmon exposed to TBT in the laboratory (1620 ng/g wet weight total Sn in exposed fish versus 40 ng/g in control fish). However, their method could not determine individual butyltin species. For comparison, the total butyltin concentration in livers from English sole captured in the Duwamish Waterway was 240 ng/g wet weight as Sn with 90% of the total present as DBT. In the Davies & McKie (1987) study, the level of total tin in the liver of exposed fish was 5.2 times greater than in the muscle. Tsuda *et al.* (1986) analyzed bass and carp muscle for TBT and DBT only, and found TBT concentrations 2 to 9 times greater than DBT concentrations (the highest TBT concentration reported was 8.7 ng TBT/g wet weight as Sn). Concentrations of TBT, DBT and MBT in homogenates of whole herring from Vancouver Harbor were 240, 50 and 60 ng/g, respectively (Maguire *et al.*, 1986). Sullivan *et al.* (1988) found levels of TBT ranging from <5 to 30 ng/g in muscle of wild caught salmon and 57 to 188 ng/g in aquaculture raised salmon. Dibutyltin was not detected in any of these muscle samples (limit of detection was <10 ng/g DBT wet weight as Sn).

Our results, showing differences between the butyltin profiles found in sediments compared to English sole livers, can be explained on the basis of the ability of fish to metabolize TBT. Lee (1985 and 1986) showed that the fish *Leiostomus xanthurus* (called the spot) was able to take up and metabolize ¹⁴C-labeled TBT from water or food to form DBT, MBT and polar products. The TBT and these metabolites were found in the intestine, liver and muscle tissues of the fish. The cytochrome P-450-dependent, mixed-function oxygenases (MFO) were suggested as being involved in the metabolism of TBT. The cytochrome P-450 MFO system is also reported to be responsible for the metabolism of alkyltins in mammals, where a sequential dealkylation pathway is observed (Kimmel *et al.*, 1977). Since TBT uptake by fish has been observed, and since fish species (including English sole) are known to possess P-450-dependent MFO systems (Buhler & Williams 1988), the finding of DBT in English sole liver from areas in which TBT was the major contaminant was not surprising.

The significance of sediment-associated butyltins is difficult to evaluate at

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present Maguire & Tkacz (1985) and Stang & Seligman (1987) report that very little TBT is released from undisturbed sediment in microcosms or *in situ*. On the other hand, Unger *et al* (1987) report that TBT is both adsorbed and desorbed by three Chesapeake Bay sediments with the sorption and desorption coefficients (liters/kg) for TBT being approximately equal. The concentration of TBT in the Duwamish Waterway sediments was more than 10^5 times greater than water levels that have been found to produce detrimental effects in oysters (Hall & Pinkney, 1985). Thus, if desorption of butyltins from sediment occurs at all, it is possible that bottom-dwelling organisms, including fish, could be exposed to harmful concentrations of TBT in water near the sediment-water interface. Maguire & Tkacz (1985) also found that oligochaete worms can take up sediment-associated TBT, thus benthic organisms may be another potential route of butyltin exposure for bottom-feeding fish.

In conclusion, a comprehensive and reliable method which included QA procedures was developed and used to analyze the four butyltin species in sediments and tissues. The method will help further our knowledge of the presence, fate and effects of butyltins in the environment. The results showed that butyltins have found their way into the marine environment and resident aquatic life of Puget Sound. The information from this initial survey points to the need for future expansion of these investigations in other areas of Puget Sound and other estuaries in the US. Moreover, with this method, uptake, metabolism and cause and effect relationships can be better studied.

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